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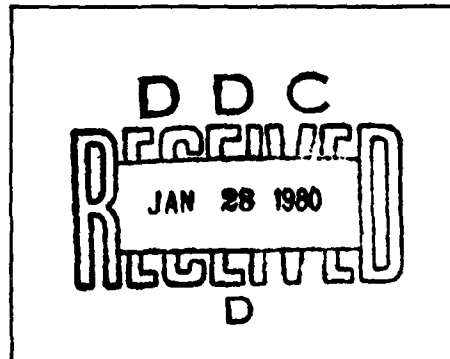
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STRENGTHENING OF METALS BY MEANS OF FIBERS
(Chapter 6 and 7)

by

V. S. Ivanova, I. M. Kop'yev, et al

EDITED TRANSLATION

FTD-ID(RS)T-0224-79   3 April 1979

MICROFICHE NR: *ꝙD-79-C-000501*

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English pages: 68

Source: Uprochneniye Metallov Voloknami, Moscow,
1973, pp. 167-204.

Country of origin: USSR
Translated by: LINGUISTIC SYSTEMS, INC.
F33657-78-D-0618
John Mose
Requester: FTD/TQTA
Approved for public release;distribution unlimited.

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U. S. BOARD ON GEOGRAPHIC NAMES TRANSLITERATION SYSTEM

Block	Italic	Transliteration	Block	Italic	Transliteration
А а	*А а*	A, a	Р р	*Р р*	R, r
Б б	*Б б*	B, b	С с	*С с*	S, s
В в	*В в*	V, v	Т т	*Т т*	T, t
Г г	*Г г*	G, g	У у	*У у*	U, u
Д д	*Д д*	D, d	Ф ф	*Ф ф*	F, f
Е е	*Е е*	Ye, ye; E, e*	Х х	*Х х*	Kh, kh
Ж ж	*Ж ж*	Zh, zh	Ц ц	*Ц ц*	Ts, ts
З з	*З з*	Z, z	Ч ч	*Ч ч*	Ch, ch
И и	*И и*	I, i	Ш ш	*Ш ш*	Sh, sh
Й й	*Й й*	Y, y	Щ щ	*Щ щ*	Shch, shch
К к	*К к*	K, k	Ъ ъ	*Ъ ъ*	"
Л л	*Л л*	L, l	Ы ы	*Ы ы*	Y, y
М м	*М м*	M, m	Ь ь	*Ь ь*	'
Н н	*Н н*	N, n	Э э	*Э э*	E, e
О о	*О о*	O, o	Ю ю	*Ю ю*	Yu, yu
П п	*П п*	P, p	Я я	*Я я*	Ya, ya

*ye initially, after vowels, and after ъ, ь; e elsewhere.
When written as ё in Russian, transliterate as yё or ё.

RUSSIAN AND ENGLISH TRIGONOMETRIC FUNCTIONS

Russian	English	Russian	English	Russian	English
sin	sin	sh	sinh	arc sh	sinh⁻
cos	cos	ch	cosh	arc ch	cosh⁻
tg	tan	th	tanh	arc th	tanh⁻
ctg	cot	cth	coth	arc cth	coth⁻
sec	sec	sch	sech	arc sch	sech⁻
cosec	csc	csch	csch	arc csch	csch⁻

Russian	English
rot	curl
lg	log

Chapter 6

Technology of Production of Composite Materials

1. Classification and Basic Stages of Development of Composite Materials

Production of fibrous composite materials with metallic dies is accomplished by various technological methods including indirect methods /1/: 1) hot drop forging, 2) impregnation by molten metals, 3) electroplating, 4) plasma plating, 5) plating from vapors, 6) cold pressure forging and sintering (powder metallurgy), 7) extruding and rolling (with supplementary annealings), 8) bonding the composite components by a pulsed energy feed.

Direct methods of producing composites include: 1) directional eutectoid conversion, 2) eutectic crystallization, directional growing of dendrites, 3) creation of fibrous structures by deformation of two-phase systems.

From the economic point of view, the indirect methods presently appear   to be more promising since, in the process of their realization, there are extensive possibilities for improving the technology of composite materials. Among the indirect methods, most attention is given to the hot forge pressing method as the "softest" method with possibilities for extensive regulation of the parameters of the technological process (time, temperature, pressure). Any method of production of composite materials should ensure 1) production of the required shape of material, 2) introduction of reinforcing fibers into the die without causing fracture, 3) absence of technological interaction of the filler materials and the die, 4) achievement of the required strength at the fiber-die interface, 5) possibility of introduction of significant strain hardening fibers which

1

cause hardening of the volume fractions and 6) uniformity of distribution of fibers.

The technological process of producing composite materials by indirect technological methods involves the following basic stages: 1) preparation of components of the composition (fibers and die) for bonding, 2) "assembly" of the composition, 3) process of bonding the components, 4) final (finishing) operations -- heat treatment, trimming etc.

There presently are very few studies which establish the connection between the technological parameters of the process, the properties and state of the starting materials and the properties of the composites produced /1/.

The development of any technology of a composite material includes several sequential stages /1/.

1. Determination of the technological interaction of the fibers and the die/1/.

2. Achievement of geometrically regular distribution of the fibers in the die.

3. Achievement of a strength of the composition near to that designed according to the law of mixtures.

4. Reduction of dispersal of properties of the composition.

5. Increase of the sizes of composite materials produced and achievement of diversity of shapes of articles.

6. Reduction of cost of production of the material.

Evaluation of the interaction and "stability" of the
fibers in the die very likely has the greatest significance
in development of a technology of composites. The only
effective method of control of the technological interaction
of the fibers in the die is control of the mechanical pro-
perties of the fibers before and after introduction of them
into the die of the composite material. The most diverse
methods (light microscopy, electron microscopy, micro-x-ray
analysis, diffraction of electrons, microhardness) were used
for evaluating the interaction of the fiber and the die but
none of these complex methods provided a clear-cut evaluati-
on of the initial moment of deterioration of the properties
of the fibers during creation of composites.

In order to achieve the designed strength of the com-
posite, it is necessary to ensure adequate strength at the
fiber -- die interface which ensures transfer of loading to
the fiber.

Fractographic studies of surfaces of fracture and ana-
lysis of fractionation of fibers under tension indicate
the positive effect of a stronger bond; only a strong bond
at the fiber -- die interface guaranteed high elastic
characteristics of the composite material.

However, on the other hand, production of a strong
bond is associated with development of diffusion processes
at the interface which leads to reduction of strength of the
fibers and to reduction of strength of the composition. In
a word, the evident presence of a connection between the
strength of the composition and the strength of the bond at

3

6. Reduction of cost of production of the material.

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In order to achieve the designed strength of the composite, it is necessary to ensure adequate strength at the fiber — die interface which ensures transfer of loading to the fiber.

Fractographic studies of surfaces of fracture and analysis of fractionation of fibers under tension indicate the positive effect of a stronger bond; only a strong bond at the fiber — die interface guaranteed high elastic characteristics of the composite material.

However, on the other hand, production of a strong bond is associated with development of diffusion processes at the interface which leads to reduction of strength of the fibers and to reduction of strength of the composition. In a word, the evident presence of a connection between the strength of the composition and the strength of the bond at

the interface of the components has been little studied
experimentally. Specifically, the optimal ratio between
the strength of the bond to shear and to brittle fracture
for each composition still remains unclear. These studies
are still in the initial stage. Even now, many compositions
produced by different methods have reached the third stage
of development (according to the list above) and development
of aluminum -- boron composite material produced by hot
press forging has reached the fourth stage. Studies at the
level of the fifth stage are now being conducted for this
material.

The problem of reduction of cost of composites (sixth
stage) is still a matter for the future.

Table 1 /1/ presents basic composite systems, during
production of which it was possible to maintain high initial
strength of the fibers. Upon examination of table 1, the
first thing to attract our attention is the comparatively
large number of composites, the technology of production of
which already has been developed for samples and the small
number of dies (Al, Mg, Ti, Ni, W, Ta, Nb, Cu) for which
this is the case. The most applicable of dies being rein-
forced is aluminum: reinforcement of aluminum was realized
by practically all technological methods and the largest
number of finished compositions (8) also involves aluminum
and aluminum alloys.

It is presently possible, for some composite materials,
to develop not only a technology of production of samples
but also a technology of production of different intermediate
products and articles.

Table 2 /1/ presents basic achievements of technologists
in development of composite constructions.

Data from table 2 show that intermediate products and
articles made from composite materials presently do not have
the diversity required. This situation, however, is tempor-
ary and intensified studies will lead to a large increase
in diversity of shapes.

Table 1
Technology of Production of Composite Materials /1/.

No.	TECHNOLOGY	DIE - FIBER COMPOSITE MATERIAL
1	IMPREGNATION BY MOLTEN METAL	STEEL Al—B, Al- , Al--W, Mg—B, Ni—W, Cu—W, Cu—Mo
2	HOT DROP FORGING	Al–B, Al—Be, Al—SiC, Al–B COATED Al), Al—SiO₂, Mg—B. Ti–TiC, Ti—Be, Ti –B, Ti—B
3	COLD PRESSURE FORGING AND SINTERING	COATED Al) Ni– W, Ni –Mo, Ti—Mo, Ag—W
4	PLASMA VAPORIZED COATING	Al –B, Al—SiC, Al—B COATED Al), W—W
5	SHAPING WITH HIGH SPEEDS OF ENERGY INPUT	Al –B, Al—W, Ni—B, Ni—W, Ni—SiC, Ti—B, Ti—TiC
6	ELECTRODEPOSITION	Al –B, Al—SiC, Ni—B, Ni—SiC, Cu—W, W—W
7	CHEMICAL DEPOSITION	Al—Be, W—B, W—W
8	EXTRUSION AND ROLLING	Al – B, Ti—Mo, Ti –B, Ni—W, Ni—Mo
9	DIRECTIONAL EUTECTIC CRYSTALLIZATION	Al—Al₃Ni, Cu—Cr, Al—CuAl₂, Sn—Cu₆Sn₅, Ta—Ta₂C, Nb—Nb₂C

11. Indirect Methods of Producing Compositions

Hot pressure forging. The hot pressure forging method has been used for production of composite materials from aluminum, titanium, magnesium and nickel dies.
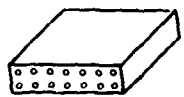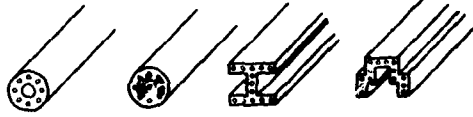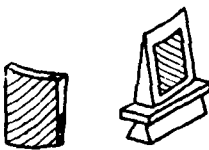
Blanks for subsequent hot pressure forging were produced most often by winding on a drum with flat surfaces coated by foil. The fiber is fixed in the prescribed position by a bonding agent which is removed in the hot pressure forging process. In some cases /2/, fibers of the die material, which separate the fibers of the hardener, are used to produce strictly regulated distances betwen fibers during winding.

In special cases, reinforcing materials, lain between foil sheets, may be in the form of an ordinary network containing longitudinal and transverse fibers from hardening material or in the form of a complex network consisting of longitudinal reinforcing fibers reinforced by the transverse fibers from die material.

The blanks produced are placed in stamps and then are pressed into composite material: the pressure, time and temperature of bonding are varied. In order to produce compositions with precision distribution of the reinforcing phase, low pressures are used and the temperature and time of action of the pressure are increased. Some data /2/ indicate that, during pressure of the order of one atmosphere, it is possible to produce by hot pressure forging (diffusion welding) not only intermediate products but also articles in the form of honeycomb panels. Hot pressure forging may be performed in air, in a vacuum or in an inert gas medium, depending on the material being used.

Table 2

Kinds of Articles and intermediate Products Produced by
Different Methods of Production of Composite Materials /1/

1 Вид материала	2 Схема продукции	3 Методы получения
4 Однослойная лента		10 Динамическое прессование, электроосаждение, горячее прессование
5 Армирование труб по окружности и по образующим		11 Динамическое прессование, напыление из газовой фазы, прессование, пропитка
6 Листы и плиты		12 Горячее прессование, динамическое прессование
7 Конструкционные профили		13 Литье, экструзия
8 Сотовые панели		14 Горячее прессование (диффузионная сварка)
9 Конструкционные армированные вставки		15 Деформирование армированных листов, получаемых горячим прессованием

Key:
1- Kind of material, 2- Diagram of the product, 3 - Methods
of production, 4- single-ply strip, 5- Reinforcement of
tubes along the circumference and along the generatrix,
6- Sheets and plates, 7- Structural shapes, 8- Honeycomb
panels, 9- Reinforced structural mountings, 10- Dynamic
forge pressing, electrodeposition, hot forge pressing,
11- Dynamic forge pressing, deposition from the gaseous
phase, bonding, impregnating, 12- hot forge pressing, dynam-
ic forging, 13- Casting, extrusion, 14- Hot forge pressing
(diffusion welding), 15- Deformation of reinforced sheets
by hot forge pressing.

The use of single-ply, narrow-strip, semi-finished products (fig. 1,b) for hot forge pressing provides, by means of alternating the direction of fibers in the layers, ~ot only good orientation of unidirectional composites but also two-dimensionally reinforced material.

The method of hot forge pressing of fibers, pre-coated with die material (see fig. 1,v) was used successfully for production of composite material in an Al -- SiO_2 system. The use of this technology requires the use of soft readily deformable dies for good distribution of fibers. The most laborious of all methods of hot forge pressing is the method of impregnating a beam of fibers by a powdered die (see fig. 1,g). After the preparatory operation (priming the positioned fibers with the powder die) the operation itself of hot forge pressing (isostatic hot forge pressing or ordinary pressing in dies) follows.

Impregnation by molten metal. Methods of impregnation of fibers by molten metals may be extremely diverse : 1) impregnation of a beam of fibers due to capillary forces, of forced pressure of the column of molten metal due to atmospheric pressure; 2) pulling a beam of fibers through molten metal; 3) forced impregnation by molten metal due to creation of high pressures (centrifugal method, casting under pressure).

Methods of impregnation by molten metal have been used only in case of the absence of noticeable interaction between the components of the composition. Evidently, the first composite material produced by this method was a Cu-W composition.

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Fig. 1. Diagrams of Production of
        Fibrous Composite Materials

a- hot pressure forging, b- hot
pressure forging of single-ply
semifinished products, c- hot
pressure forging of fibers coated
by dies, d- dross casting /1/;

1-foil, 2- fiber, 3- die,

4-single-ply semi-finished

product, 5- dross from powder,

6- network

9
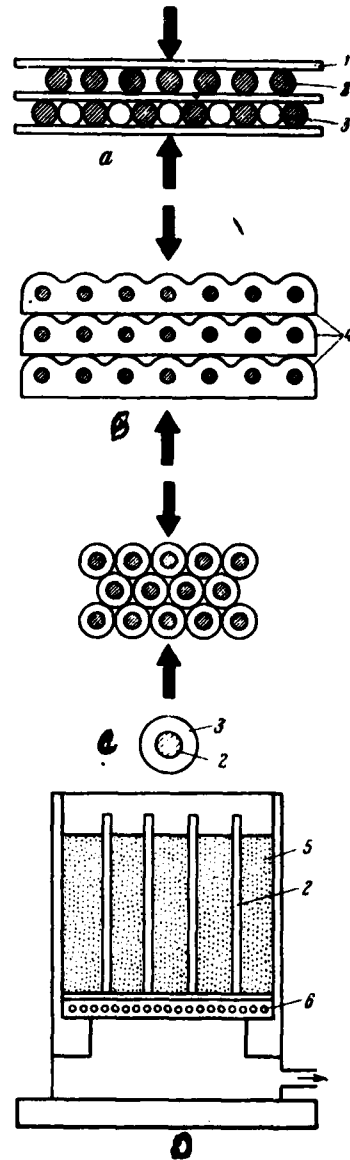
This method has definite promise of development for reinforcing a metal die by ceramic fibres (Al_2O_3, SiU and others) while most of the prospective fibers (especially boron and carbon fibers) interact strongly with molten metals and thus limit the practical possibilities of this method.

Of all kinds of promising compositions, only Mg - B composition has been used successfully in an impregnation method to produce samples with compressive strength up to 245 kg/mm^2/1/. However, there is reason to assume that the impregnation method will be used more widely. The use of boron fibers with coatings (SiU, bN and Ag) greatly reduce softening of the reinforcing phase in the impregnation process /1/. Figure 2 gives a schematic presentation of some varieties of the impregnation method.

The method of drawing fibers through melts (see fig. 2,a) has been useful and effective in some cases as, for example, for compositions of Al -- carbon fibers where, by means of reducing the time of passing the fibers through the melts by several seconds, it was possible to incur only slight softening of the fibers.

While vacuum impregnation of a beam of fibers by molten metal does not, on the whole, provide, for many metals, good results (there is incomplete and uneven impregnation) it did provide, in the case of Mg - B composition, production of bars of a composition with 75% by volume of B and with hexagonal tight packing of the fibers /1/. In this version (see fig. 2,b,c), it was possible to obtain composite materials with unidirectional fibers in any shape of cross section: bars, tubes and different kinds of structural shapes (channel bars, brackets, T-shapes and others).

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Such unidirectional shapes have high specific strength $(5 \cdot 10^4 \text{m})$ and specific rigidity of $\sim$ 1260 k.
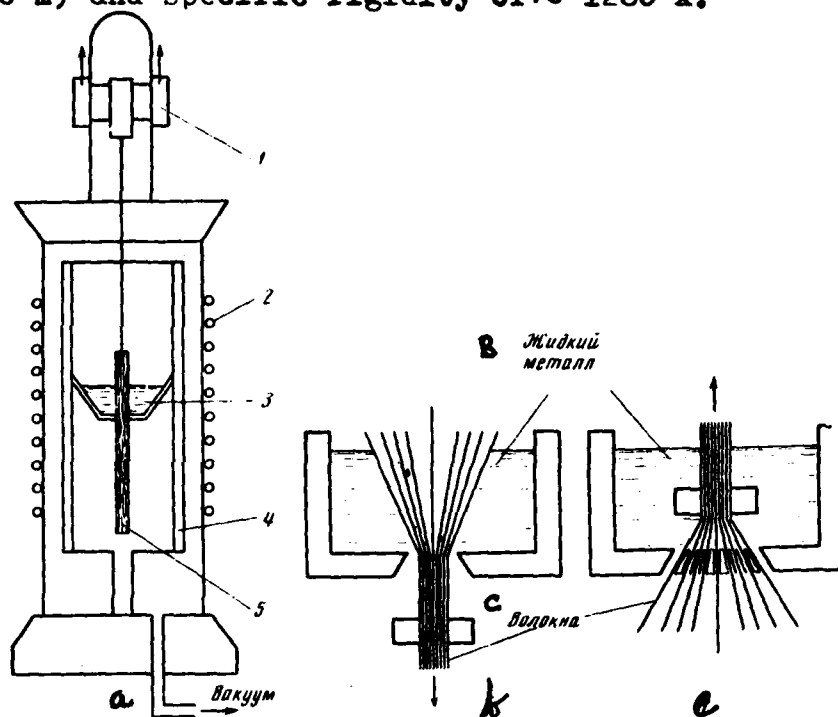
Fig. 2. Diagrams of Production of Compositions By Means of
Liquid-phase technologies /1/

a- vacuum impregnation of a beam of fibers, b,c- shaping a
bar of composite material by passing a beam of fibers through
a bath

1- magnet, 2- furnace, 3- melt, 4- graphite

a- vacuum, b- liquid metal, c- fiber

Recently/3/, definite successes have been achieved in
the impregnation under pressure of a felt of thin (0.1-2мк)
whiskers by smelted metals (ERDE-process). Aluminum alloys
were used as a basic impregnating materials and felt from
silicon carbide and silicon nitride whiskers was used as a
strengthener. Within the rather short duration of the pro-
cess (less than 1 second), there was successful production

of a composition from nominally incompatible materials
(whiskers of silicon nitride and aluminum, for example).
The best results are obtained for compositions of an alumi-
num alloy -- 21% by volume of silicon carbide whiskers. De-
tailed data concerning this material have not been reported
but there has been mentioned that, in comparision with a
die, this material had twice the modulus of elasticity and
higher strength properties, including a higher failure
strength.

Electrolytic deposition. This method of producing com-
posite materials may be used successfully for preparation
of compositions with components which interact at high tem-
peratures. The essence of the method involves electrolytic
deposition of material of the die either on a frame with
stretched fibers or on special mandrels on which the die
is deposited and the fittings are coiled simultaneously
(fig. 3). This method of producing composite materials was
tested by Bonpano and Beyker /1/ and is applicable, in prin-
ciple, for any compositions , the die of which may be de-
posited from an electrolyte and it has the following advan-
tages: 1) the process is conducted at room temperature;2) the
method permits production of electrolytically deposited
samples of a die with density near to the theoretical densi-
ty; 3) the method ensures good contact between the fiber and
the die; 4) the method may be used to produce articles of any
shape which represents a solid of rotation; 5) it is possible
to control precisely distances between fibers i.e. the
volume fraction of the strain hardening phase.

A fault of the method is the formation of pores during
butt-jointing of layers, rising from adjacent fibers and the
backing. This phenomenon occurs especially during large

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volume fractions of fibers. The second cause of the formation
of pores is the incompatability of shape of the surface of
the first layer of fibers coated by the die and the shape
of the strain hardening fibers. We must emphasize that, even
in cases in which the shape of the surface ensures stacking
up of the second layer of fibers without formation of pores,
the nature of the bond between the fiber and the die at the
point of contact differs from the nature of the bond in the
other parts of the circumference of the fiber.
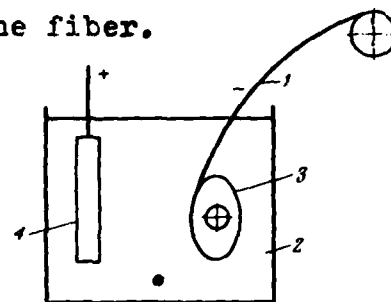
Fig. 3. Diagram of Production of a Composite material by
Deposition of the Die on the Fiber in an Electroly-
zate /1/

1- fiber, 2- bath with salt solution, 3- cathode-mandrel,
4- anode

Basic results of technological studies of production of
composite materials by the method of electrolytic deposit-
ion indicate the following /1/.

1. Strips with one layer of fibers and containing a
minimal number of pores may be produced up to 45 percent by
volume of the hardening phase.

2. During production of multilaminar compositions, the
number of pores increases and, in order to reduce their num-
ber, it is necessary to overcome serious difficulties in-
cluding the failure to maintain a definite rate of deposit-
ion for each volume fraction.

3. In order to improve the quality of composites pro-
duced by the method of electrolytic deposition,it is
necessary to compress them.

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4. Multi-ply composite materials may be produced from single-ply strips produced by electrolytic deposition by means of joining them by hot drop forging.

The method of electrolytic deposition may be used to produce multi-ply composite materials which possess high strength: an Al - B composition with 70% B by volume (10 layers) displayed a strength of $\sim$140 kg/mm^2 with a modulus of elasticity of 24,000 kg/mm^2.

Plasma metal spray coating. The plasma metal spray coating method has been used for Al - B and W - W compositions. A diagram of the plasma method of producing composite materials is shown in figure 4.

Metal spray coating is conducted in a chamber containing an inert atmosphere or with the use of a protective jet of flame. The bed of the die is preliminarily dusted on a mandrel and ,then, the mandrel is wound round with reinforcement iron and another layer of the die is sprayed on the reinforcement **fiber**. A die produced by the method of plasma metal spray coating has considerable porosity (up to 15 percent by volume), a high level of oxides (up to 1.5 percent by weight) and correspondingly low plasticity in comparison with ordinary material of the die.
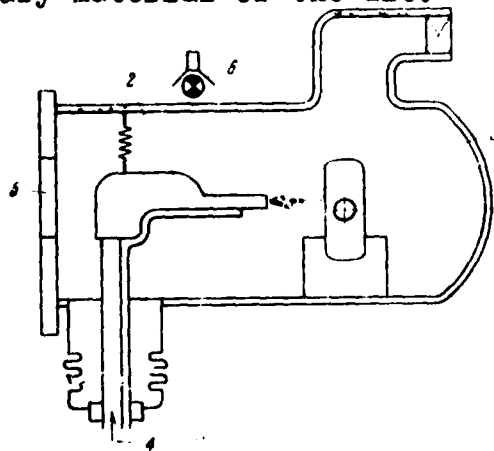

Fig.4. Diagram of the of Plasma Method of Producing Composite Materials /1/

1- Chamber housing, 2- Plasma jet, 3- Mandrel for composites, 4- Feed of water, powder, power, 5- Observation window, 6- lamp

It is true that reports concerning possibility of producing an almost non-porous die by the plasma method have appeared recently /4/.

In an ordinary case, multi-ply compositions produced by the plasma metal spray coating method require supplementary sealing, only after which the die of the composite material achieves approximately half the strength of an ordinary die. In the process of plasma metal spray coating, the strength of the reinforcing fibers are, as a rule, reduced. Thus, for example, the strength of boron fibers was reduced by 20% during production of an Al – B composite.

Recently, there has been success in the almost complete avoidance of loss of strength in reinforcing fibers by means of the use of boron fibers coated by silicon carbide which is less sensitive to a high level of oxygen in a plasma – coated die. Although production of composite materials by the plasma metal spray coating method is more expensive than other methods, it, at the same time, has one indisputable advantage in the possibility of direct production of articles from composites.

Chemical deposition from vapors. This method of production of composites is only now being developed. The first experiments, conducted on an Al – Be system (decomposition of aluminum acryls on a heated mandrel containing berillium wire), showed its advantages (possibility of producing high compactness of die material and absence, because of the low temperature, of significant interaction between the fiber and the die) and shortcomings (formation of pores during production of multi-ply composites because of irregularities of the formed surface).

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After further studies on optimization of the process, there is a possibility of producing continuous strips reinforced by a row of fibers /1/ by the method of chemical deposition of the die from the gaseous phase.

Cold drop forging and sintering (powder metallurgy method). The use of this method for producing composite materials has not been used extensively because of difficulties associated with the uniform distribution of fibers in a powder die and the prolonged sintering process which leads to interaction of components of the composition and reduction of the strength of the fibers. This method was used, in most studies, as preparatory work for subsequent operations by hot extrusion and rolling.

Extruding and rolling. These methods of producing composites were used successfully in the creation of some composites: Al - B, Ni - W (extrusion), Al - stainless steel, Al - W. Ti - W (rolling).

The most complete study of the technology of production of composites by the extrusion method was conducted by Baski /5/ who used, for the study of this process, blanks produced by the powder metallurgy method and consisting of cobalt, a cobalt alloy, Nichrome 80-20 and stainless steel, reinforced by tungsten and molybdenum wires. This investigation began with establishment of the compatability of the alloys and the fibers, which is of interest, and this was followed by by preparation of composite materials with continuous and individual fibers , during which extrusion and hot rolling were used.

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It was demonstrated that it is possible to produce bars of composite materials of uniform cross section with unidirectional fibers from blanks of composite materials produced by the powder metallurgy method. Reinforcement of nickel and titanium alloys by refractory fibers enhances their mechanical properties and, moreover, composite materials reinforced by continuous fibers displayed superior properties than materials reinforced by individual fibers.

A variety of the extrusion method of producing composite materials is the method of pressure forging of a bundle of fibers, coated a layer of the die/6/. This method is applicable mainly to metal - metal systems while the die is simultaneously a material which protects the fiber from oxidation. Fibers, coated by the die, are extruded into a ring of the protective material (fig. 5,a). In case of incompatability of the fibers and the die (protective coating on the fiber), there is applied to the fiber an intermediate layer, the material of which is compatible with the fiber and with the die. After extrusion of such blanks, there is formed a composite material with an extremely regular, dense polygonal structure. In order to coat faces of fibers with a protective layer, it is recommended that the extrusion blanks in the ring be separated by a layer of the die (see fig. 5,b).

This method may be used not only to produce cylindrical composite materials but also to produce more complex shapes (see fig. 5,c) which approximate the shape of real parts. This method permits production of materials with the following combinations of fibers and dies:

Fibers - beryllium, molybdenum, tungsten, niobium, titanium, refractory cobalt alloys.

Dies (coating) - nickel-chrome alloys, alloys based on

hafnium, aluminum and its alloys. Experiments showed that
the optimal volume fraction of the die is ~ 25%.

In developing the method of extrusion as a means of
producing composite materials with continuous fibers, Berg-
hezan /7,8/, proposed several methods of production of ex-
trusion blanks which ensure uniform distribution of fibers
in the die (fig. 6). In fig. 6,a, the blank under compress-
ion consists of a set of coaxial cylinders in the gaps between
which are inserted alternating fibers of the materials of
the die and the reinforcer. Figure 6, b - g, presents three
different methods of forming cylindrical blanks under ex-
trusion. The use of thin foils of die material permitted
procurement of significant percentages of a hardening phase
with almost ideal disposition of fibers. We must emphasize
that blanks produced by this method (see fig. 6,g) but with-
out coiling may be used as an ordinary method of compress-
ion. The extrusion method is comparatively inexpensive in
comparison with other methods of production of fibrous com-
posite materials and does not require powerful presses. Ber-
ghezan used this method to produce compositions based on or-
dinary stainless steels, alloys of Khastela X and Ni - Al,
reinforcing them with tungsten and molydenum wires. The
maximal volume fraction of the reinforcing fibers reached
50%.

During production of compositions by rolling, there is
usually used transverse rolling (across the fiber) which
does not damage the relatively brittle reinforcing fibers
and, as reinforcement, networks with transverse fibers
from die material are used.

In producing composite materials, we also use extrus-
ion and rolling as finishing and dressing operations.
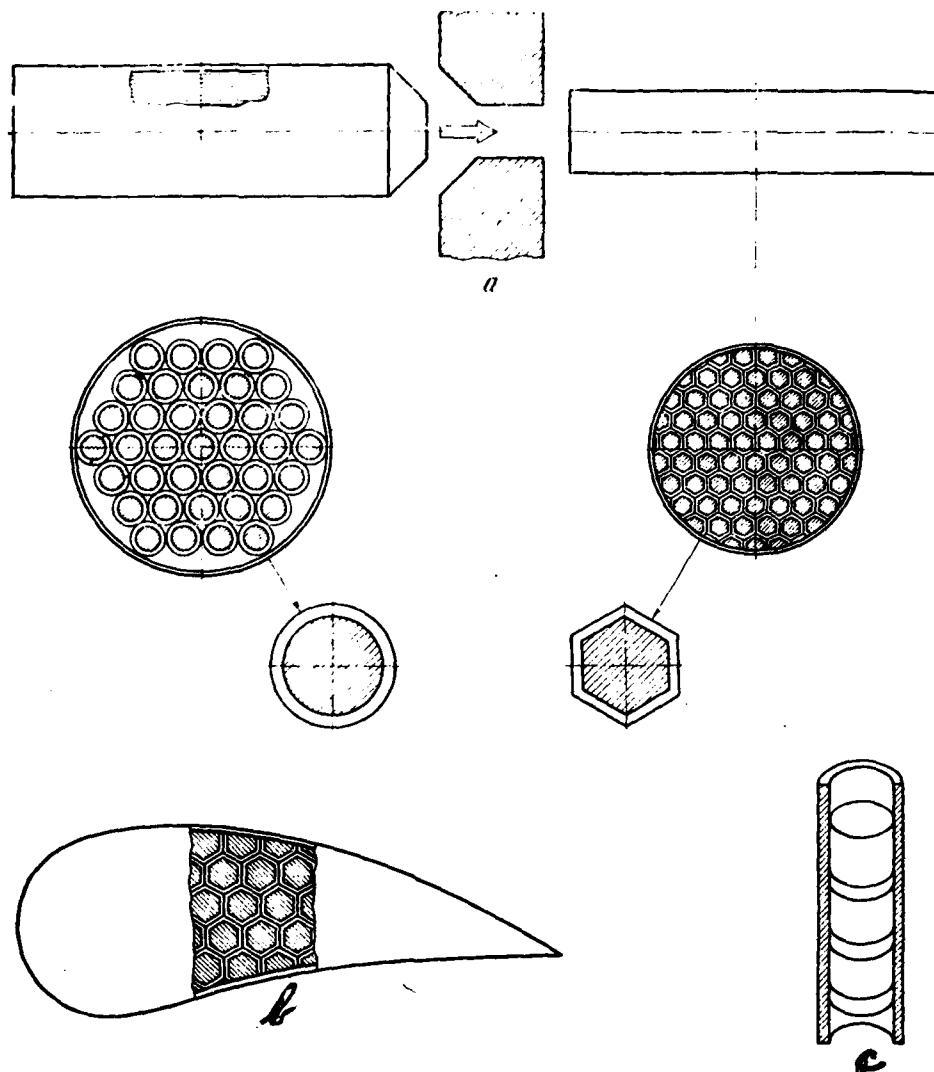
Figure 5. Diagram of Production of Composite Material by
Means of Extrusion of Fibers, Coated by Dies
a- Diagram of extrusion and structure of material
produced; b- structure of extruded shape; c- dia-
gram of assembly of extruded blanks, which permits
coating of the face of the fibers by die material
/5/.
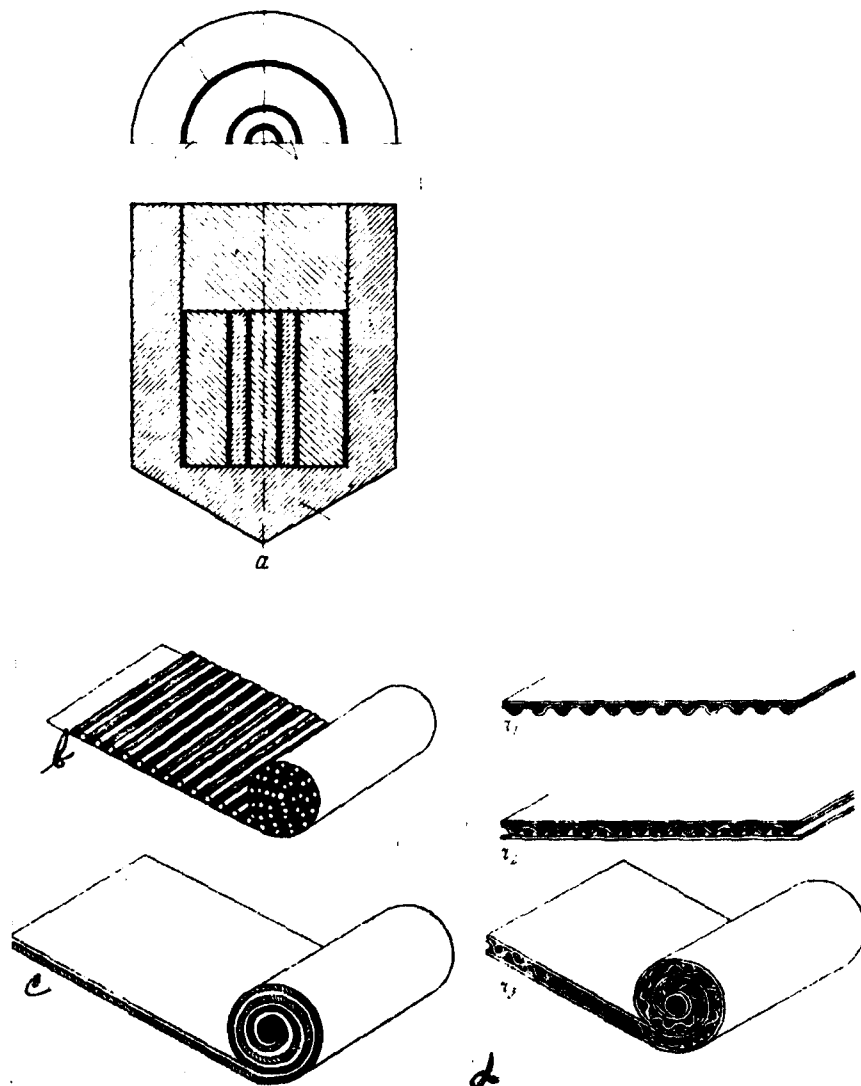
Fig. 6. Diagram of Assembly of Blanks for Production of Composite Materials by the Extrusion Method
a- The blank consists of rings of the die with reinforcing fibers applied between them;b,c,d- diagrams of assembly by means of twisting blanks from fibers of the die (white) and fibers of the strengthener (black) and also layered blanks /7/.

High-speed methods of bonding components of compositions. In some studies of production of composite materials, percussion was used for bonding components of compositions. At Battelle Institute /1/, some composites, including Ti - SiC system, were produced by use of a pneumatic percussion machine which produces pressure pulses of up to ~ 280 kg/mm^2. As a rule, the pulsed pressure was applied to mixtures of powders of the die and fibers enclosed in a shell and heated to high temperatures. For some compositions, including Ti -SiC systems, optimal conditions (temperature, pressure, time) were found for the least damage to the fibers. For this system, the temperature of pulsed casting is 1100°C (in comparison to 900°C for hot forge pressing of an analogous composition).

Great promise is shown in the use of the energy of an explosion for producing the most diverse compositions, including some based on aluminum. Selection of technological parameters for this method of producing composites is laborious but the advantages are extremely attractive: 1) there is a possibility of producing large sheets and other articles; 2) there is almost complete absence of technological interaction and reduction of strength of the fibers.

A shortcoming of the method is the almost total impossibility of using it for brittle fibers which possess great toughness.

111. DIRECT METHODS OF PRODUCING COMPOSITES

The indirect methods of producing fibrous materials examined above are used to produce artificial composite materials. There are also direct methods of producing fibrous structures by natural means which methods involve either a directional deposition of the second phase or the imparting

of the fibrous shape by deformation of the second phase.
These methods of producing fibrous materials are based, to
some extent, on the long established webb and Fordzheng /9/
experiments which demonstrated that acicular depositions of
the second phase in alloys have high strength comparable
to that of high-strength filamentary crystals.

An advantage of direct methods is the combination, in
one operation, of the production of fibers and the preparat-
ion of compositions. Their shortcomings include the limited
volume fraction of the strain hardening phase and its de-
pendence on the chemical composition of the alloy, the low
productivity of the methods, difficulties associated with the
proper geometry of the fiber. We will examine methods of
producing fibrous compositions by direct methods in more
detail.

1. The fiber in the alloy may be formed in the solid
state by eutectoid conversion and disintegration of super-
saturated solid solutions. Precipitations of the second
phase in the form of spicules were observed repeatedly in
Al - Mg -Si and Al - Mg - Cu systems /5/. However, the
volume of the precipitation phase was small ($\sim$ 5%); the
spicules were arranged in three different directions which
was ineffective in increasing the strength. There still are
no suitable systems for producing significant strengthening
in them due to precipitation of fibers of the second phase.
In steels and other alloys with eutectoid transformation,
it is possible to produce two-phase structures with marten-
site fibers. One of the means of producing such a structure
is the method of partial quench-hardening. Hypoeutectoid
steel is rolled at a temperature above Ac_3 and a fibrous
structure of austenite in ferrite is produced. After quench
hardening of the material, its structure consists of ferrite

and austenite in the form of fibers. Kerans /5/ conducted similar processing of carbon steel with 0.2% C and increased the strength from 42.8 to 105 kg/mm^2.

2. Fibrous structures from a melt may be produced by regulating the process of solidification by two methods: directional crystallization of the eutectic and directional growth of the dendrites. Crystallization of the eutectic during directional heat removal leads to formation of directional fibrous stuctures /10/. Directionality of crystallization may be realized, for example, by slow withdrawal of the crucible with the heated metal from the heated zone of the furnace or by means of zone refining. The possibility of producing directional structures by the eutectic crystallization method was studied mainly for Al – Ni, Al – Cu, Al – Cr, Ni – B, Ni –Be, Nb – C and Ta – C alloys /11/. In some cases it was possible, by means of directional crystallization to achieve a 3-fold increase of strength of the cast material. Thus, an ordinary eutectic of Al – Al$_3$Ni has, in the cast state, σ_b = 9 kg/mm^2, $\delta \sim 15\%$ and that with a fibrous structure σ_b = 30 ÷ 40 kg/mm^2 and δ = 2% /12/.

Fig. 7. Sequence of changes of structure of two-phase material being transformed during deformation in a fibrous composite material /7/
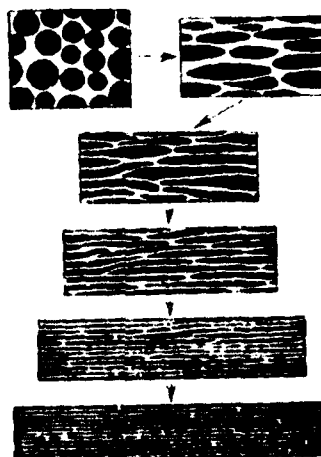
A basic difficulty in production of fibrous structures by the method of directional growing of crystals involves the fact that, after primary crystallization, the forming dendrite had only one branch. An advantage of this method is the possibility of increasing the volume fraction of the hardening phase. This method of producing a fibrous structure was studied by Davis /13/, who produced a structure composed of spicules of Cu_5Sn_6 compounds in a copper die. He showed that compositions with Cu_5Sn_6 spicules have considerable strength which may be increased by heat treatment.

3. The method of producing fibrous compounds by deformation is of great interest. Realization of this method requires the presence in the alloy of two phases, one of which has great strength at room temperature and higher temperatures. By selecting conditions of deformation, inclusion of a strong second phase may be prolonged. Petty /5/, using deformation of aluminum alloys containing intermetallic compounds at temperatures above 0.6-0.7 $T_{melting}$ of the alloy, demonstarted that particles of intermetallic compounds are elongated and acquire directionality. Analogous behavior of particles of the second phase during deformation was observed by Jones /5/ in Cu - Cd, Cu - P systems which contain Cu_5Cd and Cu_3P phases. However, these studies also indicated a definite inclination of the elongated particles to spheroidization in case of holding the processed material at a high temperature.

Bergkhenzan /7/, deforming a rolled two-phase structure (tungsten spheroids in a die from an Ni - Cr - W alloy), showed that such a method may be used to produce a fibrous composite material with a high volume fraction of the

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hardening phase. Different phases of deformation of this composition are shown in fig. 7.

The principle of formation of the fibrous hardening phase directly in the die (in situ) during production of composite material has extensive potential possibilities: in favorable cases (a combination of phases with sharply contrasting moduli of elasticity) it may be used to achieve very great reinforcement. In Berghensan's experiments, deformation of tungsten shperoids in a plastic die was accomplished by rolling at room temperature. Tungstens which are brittle under these conditions are readily pulled into a fiber. A plastic high-chromium die reliably protected the tungsten fibers forming during deformation from oxidation at high temperatures. The strength of these compositions at 872°C reached 45 kg/mm^2(at this temperature, the die strength was 5 - 6 kg/mm^2).

Kvatints and Uiton,in a paper "A Study of Tungsten Alloys Containing Fibrous and Reacting Additives", presented at a conference on powder metallurgy at New York, 7 - 14 July 1965, showed the effectiveness of hardening tungsten by producing fibrous inclusions of second phases.

The method of producing fibrous composites involves introduction, by the powder metallurgy method, into the tungstens of oxides (ZrO_2, H_fO_2, ThO_2), borides, nitrides and carbides of hafnium and tantalum carbide. Then the oxides and chemical compounds were pulled along the axis of the bar in the process of extrusion at high temperatures (2000 - 2500°C) and their volume fraction in the die reached 5 - 26%. In the extrusion process, the oxides and compounds were pulled at a ratio of length to diameter equal to 13 - 23. Oxides were

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pulled most readily. Tests of the prolonged strength of tungsten hardened by this method at $1650°C$ and 5.6 kg/mm^2 stress showed that, after extrusion, the service time of tungsten increased $25 - 50$ times, especially in case of the use of refractory compounds.

Considering the overall problem of the technology of production of composite materials, we must emphasize that it is, basically, still in the state of development; there are, with few exceptions, no basic studies in a detailed investigation of the effect of technological factors on properties of materials produced.

Researchers working in the area of production of composite materials must develop materials which are 1) stable, 2) competitive in price with ordinary materials, 3) diverse in shape.

Solution of these problems requires a detailed and careful study of the technology i.e. establishment of the dependence between technological factors, properties of components of the composition and properties of the composite material. Only the establishment of detailed dependences will permit an approach to development of scientific bases of a technology of composite materials.

It is an optimal scientific technological itself which will permit the development of composite materials and production of materials with stable properties required by the needs of technology.

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Chapter 7.
INTERACTION BETWEEN COMPONENTS OF COMPOSITE MATERIALS
1. Introduction

Since, in most cases, composite fibrous material is a
thermodynamically non-equilibrium system, interaction between
componentsof the composition becomes unavoidable both during
production of the composite material and during its service
in constructions. Proper selection of components of compo-
sitions requires careful study of the chemical compatibility
of materials of the die and of the fiber. In one of their
studies, Bates, Vald and Veynshteyn /1/introduced two new
terms (thermodynamic compatibility and kinetic compatibility)
to clarify the concept "chemical compatibility". Thermodynam-
ic compatibility is a state of stable thermodynamic equili-
brium between material of the die and that of the fiber.
Kinetic compatibility is the state of metastable equilibrium
determined by different kinetic factors: the rate of diffus-
ion, the rate of dissolution, the rate of growth of new
phases of interaction etc.

Thermodynamic compatibility of components is considered
in phase equilibrium diagrams which play an expecially im-
portant role in the production of heat-resistant composite
materials. Although CONSTITUTION diagrams are not of decisive im-
portance for thermodynamically unstable systems. they do
indicate the type of reactions and their directionality.

This interaction along the interface of components of
the composition is, on one hand, necessary, since it is the
very thing that ensures the required force of bond necessary
for transmitting the loading from the die to the fibers and
the high strength and elastic properties of the composition;

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while, on the other hand, excessive interaction along the interface leads to formation of defective layers and to deterioration of properties of the composition. Evidently, problems of compatibility of the die and fibers are of decisive importance in the development of practical compositions. In the overall view, interaction of components may lead to a change of properties of the composition: 1)interaction changes the hardening properties of fibers; 2) it leads to a change of properties of the die; 3) the value of the strength of bond between components of the composition changes.

Although the reactions along the interface may, in principle, improve the strength properties of the composition through strain hardening of the die and the fiber (alloying, dissolution of the surface defects) and by means of increasing the strength of the bond between components, developers of composite materials most frequently encounter a deterioration of properties of the material as a result of interaction.

This deterioration of properties of composite materials most frequently is associated with softening of the fibers because of their partial recrystallization, then to embrittlement of the fiber and the die, disintegration of the interface of the composition, the rise of defective reaction zones which serves as a source of disintegration.

If, during creation of metal – metal compositions, there is usually a struggle against the interaction of components, then, during creation of metal – ceramics compositions, it is sometimes necessary to strive for ensurance of interaction between the components of the composition. Considering this

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very circumstance, it is necessary to consider metal – metal systems and metal – ceramics systems and also metal – non-metallic fibers (glass, boron, graphite) differently from the point of view of interaction.

11. metal – metal Compositions

Examination of the interaction of components in composite fibrous metal – metal material should be based upon the corresponding constitution diagram. However, during theoretical analysis of interaction, it is necessary to consider (in addition to the constitution diagram) the specific properties of the flow of surface reactions in the composite material (structure of the fiber and the die, conditions of heat transfer during formation of compositions, structure of the interface and, during solid-phase interactions, deformation of the die by the fiber etc).

The interaction of components in a copper – tungsten system is most completely and systematically studied at present. This composition, produced by the method of vacuum impregnation with the use of a pure copper die, did not reveal appreciable interaction between the components. Alloying the copper die of this composition with different active elements permitted investigation of the effect of alloying on the mechanical properties of these compositions. A similar approach to the study of interaction (a comparison of the structure and mechanical properties of compositions with identical fibers but with dies which differ in form and degree of alloyability) is extremely promising for the study of other compositions, the dies of which are usually complex alloys. Petrasek and Witon /2/ alloyed copper in a copper – tungsten wire composition with aluminum, cobalt, chromium, niobium, nickel, titanium and zirconium (table 1).

We studied the structures of the die, fibers, transit-ion layers and mechanical properties (see fig. 1) for compositions produced with different volumetric levels of fibers.

These studies indicated that:

1) alloying did not, in any case, lead to an increase of strength of the composition;

2) reduction of strength was either significant or insignificant, depending on the shape of the die;

3) differences in strength properties of the compositions are associated with changes of microstructure along the surface of the fiber - die interface.

In the compositions studied, there occurred three reactions on the interfaces: 1) diffusion penetration of the alloying element into the fibers and recrystallization of the grains at the periphery of the tungsten fibers; 2) formation of a two-phase zone on the periphery of the fiber; 3) formation in the fiber of a zone of the solid solution.

A recrystallization zone in the fiber was observed after alloying the copper die with aluminum, cobalt and 10 percent by weight Ni. The cobalt quantity (1 percent by weight), which causes recrystallization in tungsten fiber was much lower than the nickel quantity (5 percent by weight). Aluminum was most intensely diffused in tungsten. The depth of diffusion penetration of alloying elements in the fiber increased with the increase of their level in the die. In alloys with a recrystallized zone, reduction of strength from the interaction along the interface was most intense.
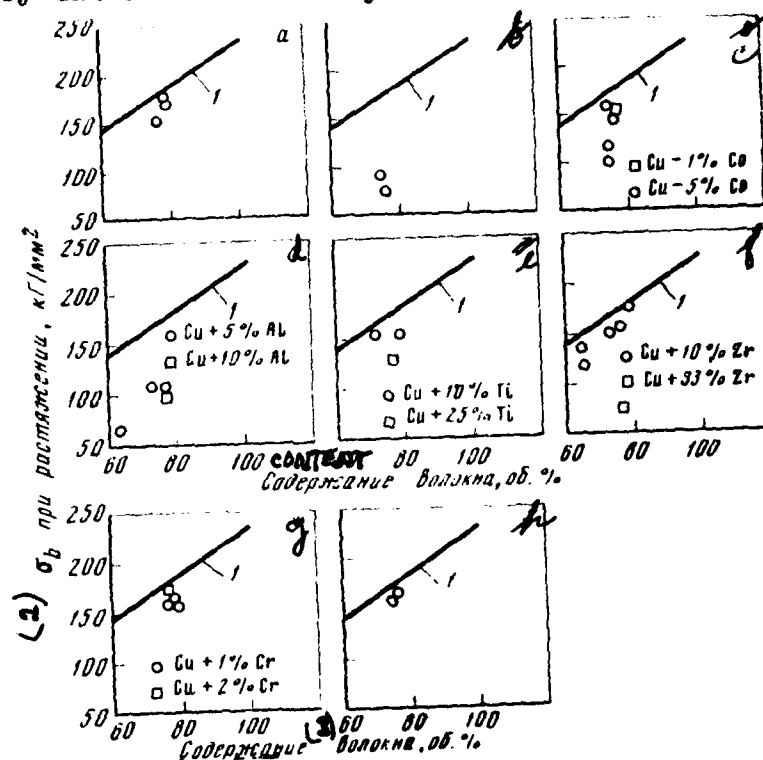
Table 1.

Mechanical Properties at Room Temperature of Alloyed Copper,
Reinforced by Tungsten Fiber /2/

1. Легирующий элемент в медной матрице	2. Максимальная растворимость легирующего элемента в вольфраме, %	3. Содержание легирующего элемента		6. Номер образца	7. Содержание волокон, об.%	8. Предел прочности при растяжении, кГ мм²	9. Относительное сужение, %	10. Характер разрушения
		4 вес. %	5 ат. %					
11. Чистая медь	19. Не растворяется	0	0		65	158		22. Вязкий
					70,2	167		»
					75,4	175		»
12. Никель	0,3	5	5,4	1	79	173	34	»
				2	78,4	175	37	»
				3	76	153	32	23 »
		10	10,9	4	74,1	92,2	0	Хрупкий
				5	75,5	76,2	0	»
				6	79,5	35,9	0	24 Полувязкий
13. Кобальт	0,3	1	1,1	7	77,3	154	—	»
		5	5,4	8	76	149	1,5	22 Вязкий
				9	74,8	160	2,3	»
				10	74,7	103	—	23 Хрупкий
				11	79,9	120	—	»
14. Алюминий	2,6	5	11,3	12	63,4	69,2	0	24 Полувязкий
				13	72,4	107	0	»
				14	76,1	108	0	23 Хрупкий
15. Титан	8	10	20,8	15	76,7	97	—	24 Полувязкий
		10	12,8	16	78,2	156	—	»
				17	71,7	154	10	23 Хрупкий
16. Цирконий	3	25	18	18	76,3	131	—	23 Хрупкий
		10	7,2	19	72,8	151	0	»
				20	78,5	179	0	22 Вязкий
				21	75,6	159	0	24 Полувязкий
				22	64,7	121	0	23 Хрупкий
				23	64,3	137	0	24 Полувязкий
17. Хром	20. Полная растворимость	33	25,5	24	75,9	74,7	0	»
		1	1,2	25	78,7	156	7,4	»
				26	77,5	160	25,8	22 Вязкий
				27	77,2	158	7,5	24 Полувязкий
18. Ниобий	21. То же	2	2,4	28	76,4	169	16,4	22 Вязкий
		1	0,6	29	75,4	166	20,6	»
				30	75,1	156	24,6	»

1- Alloying elements in a copper die, 2- maximal solubility
of the alloying element in tungsten, %, 3- level of alloying
element, 4- % by weight, 5- atomic percent, 6- sample number,
7- fiber content, % by volume, 8- breaking point during
stretching, kg/mm², 9- relative contraction, %, 10- nature of
failure, 11- pure copper, 12- nickel, 13-cobalt, 14 Al, 15 Ti,
16- Zr, 17- Cr, 18- Ni, 19- is not dissolved, 20- complete
solubility, 21- ibid, 22- viscous, 23- brittle, 24- semiviscous

A two-phase zone was observed during alloying of copper
by titanium and zirconium. The presence of intermetallic
compounds in the two-phase zone and the damage to fibers
noticeably reduced the strength of the composition. With
a high level of alloying additives (25% Ti and 33% Zr),there
was a great reduction of plasticity of the die and, in these
cases, the reduction of strength of the composition was ex-
plained not by interaction but by embrittlement of the die.

a — Cu + 5% Ni; θ — Cu + 10% Ni; c — Cu + 1 и 5% Co; d — Cu + 5 и 10% Al; e — Cu + 10 и
25% Ti; f — Cu + 10 и 33% Zr; g — Cu + 1 и 2% Cr; h — Cu + 1% Nb [2]; 1 — медная матрица

Fig. 1. Change of Strength of a Copper - Tungsten Wire Com-
position in Dependence on the Volume Fraction of the Wire
1- copper die, 2- σ_b after elongation, kg/mm^2, 3- fiber
content, percent by volume.
-- -A zone of solid solution arose after alloying the die
with chromium and niobium. Recrystallization zones did not
arise in this case. Reduction of tensile strength of the
compositions was insignificant.

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Thus, of all of the enumerated elements which are soluble in the die, aluminum cobalt and nickel, which cause recrystallization of the tungsten wire, promote the greatest reduction of strength of the composition. The harmful effect of these elements is associated with their relatively small atomic radii, which facilitate their diffusion in tungstens. These studies confirm that, at least for this composition, the solubility of the alloying elements in tungsten has a greater effect upon the strength of the composition than does the solubility of the fibers in the die.

Reduction of the strength of the composition in case of the emergence in the fiber of a recrystallized zone may be explained by two causes: 1) reduction of the high-strength section of the fiber; 2) embrittlement of the recrystallized zone which, during elongation, fails first and acts as a singular notch which leads to a more intensive drop in the strength of the fibers.

Reduction of strength because of interaction along the interfaces was observed during alloying of complex heat-resistant alloys (Rene 41, cobalt, cobalt alloy L-605, Nichrome) by wire from tungsten and molybdenum: not one of the compositions surpassed the strength of the unalloyed die at room temperature. Baski /3/ attributed the deterioration of properties of the materials created to the formation, along the interface of components of the composition, of brittle layers which reduce the strength of the fibers. It must be emphasized that, while tests at room temperature indicated significant reduction of strength of the composition as a result of interaction along the interface, high-temperature tests even revealed an improvement of properties of the composition.

An aluminum – steel wire composite system has been less systemically studied than the Cu – W system from the point of view of interaction at the interface. This circumstance, in particular, is explained by the fact that problems of interaction between aluminum and steel were investigated repeatedly in the pre-composite era and developers of composite materials, at first, used results of studies of interactions which occur in bimetals, during welding of heterogeneous materials and in the process of alitization. Interaction between iron and aluminum, occurring after immersion of the iron into molten aluminum /4/ (the so-called liquid-phase reaction) has been studied in greatest detail. It is well known that an aluminum coating, produced by immersion of steel components in molten aluminum, consists, basically, of three layers: 1) the outer layer, the composition of which approximates the composition of the aluminum bath; 2) the intermediate layer consisting of a combination of iron and aluminum; 3) zones of solid solution of aluminum and iron.

The behavior of aluminum coatings under the effect of mechanical loadings depended, to a great extent, on the thickness and structure of the intermediate layer. Many studies have been devoted to the growth and structure of this connective layer which is formed within a few seconds after contact with the molten aluminum. The older studies /5,6/ confirm the fact that the intermediate layer between the steel and aluminum consists of $FeAl_3$ compounds or of a mixture of Fe_2Al_5 and Fe_2Al_7 compounds. The most recent roentgenographic studies of the intermediate layer indicated that the brittle intermediate layer consists of Fe_2Al_5 compounds. This connective layer is separated from the iron by a thin layer, the nature of which is still not understood completely. On the basis of several studies /7/, we may assume that this

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layer consists of a solid solution of aluminum in alpha-iron.
As indicated above, the intermediate layer exerts a direct
effect on the behavior of such composite material during
deformation. In view of the high brittleness of this layer
during deformation, it is always necessary to consider the
possibility of the spread of cracks to the entire aluminum
layer. In order to avoid failure of the coating, it is
attempted to make the intermediate layer as thin as possible.
There is the opinion that, at a thickness less than 5-7 mk,
the intermediate layer does not affect the strength of the
alitized material. In order to regulate the thickness of
the intermediate layer, the following measures were proposed
(these measures are used for fibrous aluminum - steel compo-
sitions) : 1) regulation of the temperature of the bath and
of the time of interaction between the steel and the liquid
aluminum; 2) alloying the aluminum with additives which re-
duce the rate of growth of the intermediate layer; 3) a pre-
ventive coating of the steel surface with metals or compounds
which prevent interaction between the steel and the aluminum.

The dependence of the growth /8/ of the intermediate
layer from Fe_2Al_5 on the time at a constant temperature
follows a parabolic law which means that the increase of
thickness of the connective layer proceeds very quickly at
first, but, after prolonged interaction, it slows somewhat.
The dependence of the thickness of the intermediate layer on
the temperature follows a logarithmic curve. A knowledge of
these dependences permits the regulation of the thickness of the
intermediate layer by changing the time and temperature of
the interaction reaction.

Of the many studied alloying additives which reduce the
growth rate of the intermediate layer, the most favorable for

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this purpose was found to be silicon, which is added to the fusion in a quantity of the order of 12 percent by weight.

It was established that the introduction into the fusion of even 2% of silicon greatly reduces the thickness of the layer of interaction. An addition to the aluminum melt of 4 - 12% of Mg_2Si is also recommended for reducing the interaction. In order to reduce interaction between steel and molten aluminum, a preliminary coating of the steel surface with chromium, the solubility of which in aluminum does not exceed 8% at $700^{\circ}C$, was used.

Much research /7/ has involved the study of the effect of different alloying elements after their introduction into steel on the growth of the intermediate layer. These studies indicated that carbon contained in the steel prevents the growth of the intermediate layer. If the carbon is in the cementite form, there is formed a zone enriched by cementite preceding the reaction surface. The growth of the intermediate layer is slowed after alloying of the steel by chromium, copper, nickel, molybdenum and silicon. It has also been established that an increase of the oxygen level in the steel impedes the growth of the intermediate layer. Principles of growth of the intermediate layer established by researchers, with certain corrections, may be used during development of aluminum - steel compositions, especially when a layer of the die is preliminarily deposited on the reinforcement. Several scientists /10,11/ also have studied the interaction of aluminum and iron in the solid state (the solid-phase reaction). The most systematic study was conducted by Newman and Ditrich /12/ who studied the interaction during compression in a furnace of samples of pure iron and aluminum. The research showed that the reaction between Fe and Al appears only at $590 - 600^{\circ}$ C.

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In this case, acicular crystals of the Fe_2Al_5 (η-phase) interaction phase are formed on the interface. After prolonged interaction, a zone of solid solution of aluminum in iron arose. The interaction reaction begins after a certain incubation period and ensues, at first, with increasing speed but it becomes completely extinguished after several hours.

The introduction of special markers permitted observation of the shift of the interface of components: the initial boundary between Fe and Al at the time of the experiment coincides with the boundary between Al and Fe_2Al_5. This circumstance indicates that the growth of the intermediate layer proceeds because of the diffusion of aluminum atoms through the η-phase and the iron atoms do not participate in diffusion. During such unilateral flow, there accumulates, in the boundary layer, a porosity which leads to disruption of the contact between the aluminum and the η-phase and to a slowing of the growth of the layer of the intermetallic compound. After preliminary plastic deformation of the interacting components, the interaction reaction begins even at $560^\circ C$. During very severe strain (95% reduction) the interaction temperature was reduced to $400^\circ C$.

Regularities of growth of the layer of the intermetallic compound, found during studies of the interaction of pure iron and aluminum, was confirmed, in general, during studies of solid phase interaction of alloyed steels with alloys of aluminum /13,14/. Thus, for example, studies of the effect of repeated heating on the structure of the transition zone in Kh18N10T - AD1 - AMG6 bimetal revealed the same regularities /15/ of growth as in the case of pure metals.

1. Up to 450°C, no visible changes of the microstructure of the transition zone were observed.

2. After heating the bi-metal above 500°C, there occurs a chemical reaction with formation of an intermetallic compound phase.

3. A roentgenostructural study showed that the Fe_2Al_5 phase arises first and grows quickly.

4. At 550°C, conditions are created for the growth of the $FeAl_3$ type phase.

5. Microroentgenospectral analysis permitted establishment of the fact that , in the formation of the intermetallic compound phases, there is active participation of the basic components of the steel - chromium, nickel and titanium, forming intermetallic compounds $(Fe_{1.5} Cr_{0.5} Ni_{0.2}) Al_5$ and $(Fe_{0.7}, Cr_{0.2}, Ni_{0.1}) Al_3$.

6. The regularities of growth of thickness of the intermetallic compound layer are found and, for the initial stage of growth, are expressed by the formula $X = K_0 e^{(-Q/RT} \sqrt{\tau - \tau_0}$ where k_0 and τ_0 are constants, τ is the time, X is the thickness of the layer; Q, P, T are constants in the Arrhenius equation. with an increase of the time of passage of the reaction, the rate of growth of the layer of the intermetallic compound is slowed which is associated with the formation of diffusion porosity.

7. The external pressure in the roasting process inhibits the effect of reactive diffusion which weakens the bond between the layers of bi-metal. The regularities of formation of the solid-phase compound may be considered from the

position of the general theory of topochemical solid- phase reactions /16/. In this case, the bonding of the materials involves a process consisting of three sequential stages: 1) formation of physical contact (convergence of the atoms of the bonded metals) basically due to deformation (creep) of the softer metal (for example, an aluminum die) after the pressing into it of the reinforcing component; 2) activation and chemical interaction of atoms in the course of which is formed between them a metallic bond; the duration of this stage is limited by the rate of deformation (creep) in the presence of surface layers of a more solid material (the re-inforcement) since it determines the frequency of outcrop of defects (dislocations and vacancies) in the zone of inter-action and the formation of active centers; 3) heterodiffus-ion, leading to the formation and growth of intermetallic com-pound phases in the zone of the bond of the fiber and the die. Thus, the bonding of heterogeneous metals proceeds in the process of creep of both metals. The required rate of creep in the zone of contact for given materials is determined basically by three parameters : temperature, time and press-ure. The theory stated above permits calculation of optimal regimes of bonding different materials with a controlled val-ue of interaction.
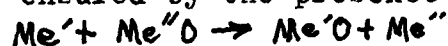
111. METAL - METAL COMPOSITES, METAL - CARBON FIBERS COM-POSITES AND OTHERS

Many ceramics are poorly soaked by metals which leads to weak bonding between components of compositions and as a con-sequence of this to weakness of the latter. Only the format-ion of a strong chemical bond between the die and fiber may ensure the designed strength of the composition. At present, much remains unclear about the nature of the rise of the

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chemical bond between the metal and the ceramics although
there are data that chemical reactions between the fiber
and the die are possible in the presence of oxygen and act-
ive alloying admixtures /17/ at least in oxide ceramics. In
this case, a transition layer, firmly bonded with the fiber
and the die, may be formed. It has been shown that, even in
the presence of an insignificant quantity of oxygen in the
Al_2O_3 - Fe composition, the reaction Al_2O_3 + Fe + O_2 ⟶
$FeAlO_4$(spinel) goes thanks to diffusion of oxygen by the in-
terface. In NiCr - Al_2O_3 vapors at $1400^{\circ}C$ there arises un-
stable CrO suboxides and, finally, Cr_2O_3. Later, at $1450^{\circ}C$,
the reaction goes up to the formation of rubidium which en-
sures a good bond of Ni and Cr with Al_2O_3.

One study /18/ presents an examination of the process of
formation of a firm bond by welding in the solid state, trans-
mitted to the metal - ceramics interface. The process is di-
vided into two stages: the stage, in the course of which,
mainly by means of creep of the metal, is formed the physical
contact of the surfaces of the metal and the ceramics and the
stage of chemical interaction which ensures the formation of
a strong bond.

The strength of the bond in a ceramics - metal composite
may be ensured by the presence of the chemical reaction
$$Me' + Me''O \rightarrow Me'O + Me''$$
which may be expressed thermodynamically in the form
$$\Delta Z^{\circ} = \Delta Z^{\circ}_{Me'O} - \Delta Z^{\circ}_{Me'O}$$

If $\Delta Z^{\circ} > 0$, interaction is impossible; if $\Delta Z^{\circ} < 0$ there
is interaction. Analysis of thermodynamic potentials of ox-
ides of metals and also of the lamination series of oxides
most frequently encountered in ceramics led to the creation

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of a scheme of appropiate selection of ceramics - metal pairs
from the point of view of their thermodynamic interaction.
This scheme may be expressed as follows:

$$\left.\begin{array}{l} CrO \\ SiO_2 \\ Al_2O_3 \end{array}\right\} +(Zr,Al,Ti,V,Ta,Nb,W,Co,Ni)$$
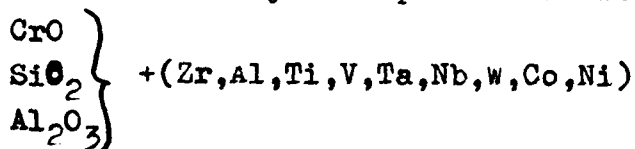
We must emphasize that this scheme of interaction is
applicable in case of conducting the interaction process in
the solid state under conditions of a deep vacuum or reduc-
ing atmosphere. For metals which do not have any natural ox-
ides, creation of an adequate bond requires additional chemi-
cal of mechanical activation.

Non-oxide ceramics, as a rule, react actively with me-
tals and, in this case, there is practically no problem in
creating a bond. The interaction of different ceramics with
refractory metals is seen clearly in table 2.

Interaction of ceramics with metals were studied in
greatest detail during development of metal compositions,
reinforced by ceramic whiskers. Results of analysis of the
thermodynamics of reactions proceeding between Al_2O_3 and
different metals are presented in fig. 2 /20/. A change of
free energy with temperature indicates, in the case of solid
phase reactions, the possibility of the proceeding of react-
ions and their directionality. The thermodynamic approach to
the evaluation of interaction must not be overestimated since
this analysis does not take into account the kinetics of the
passage of the reactions which also may be of decisive sig-
nificance in some cases of interaction of components. In
addition to this, in order for thermodynamic analysis to
give correct results, it is necessary to describe the inter-
action reaction properly. Errors in description of react-
ions greatly distort results of the thermodynamic approach

to problems of compatibility of components. According to the material above, experimental study of interaction reactions plays a basic role. Amstrong /21/ studied interaction reactions on the interface between alpha-Al_2O_3 single crystals and different nickel alloys. The change of interphase energy after alloying nickel with titanium and chromium was studied on a special vacuum apparatus by the sessile drop method. It was established that the strength of bonding between the sapphire backing and the metal drop depends on the interaction of atoms of the excess active elements,which are concentrated long the metal - ceramic interface, with aluminum oxide. An indication of interaction was the reduction of the angle of wetting of the sessile drop.

X-ray diffraction analysis revealed, for the nickel and tantalum alloy, the presence of titanium oxide at the ceramic-metal interface. X-ray methods did not reveal any signs of interaction for a nickel - chromium alloy.

Satton /2/, in an application to nickel - aluminum oxide compositions, also systematically studied the effect of additions of chromium, titanium, zirconium, copper, indium to nickel on the wettability, interaction and resistance to shearing along the interface in a nickel - aluminum oxide system. After alloying nickel with titanium and zirconium, there were obvious signs of interaction of the drop of the binary alloy of nickel and the aluminum oxide backing. Satton and Fayngold /2/ later proposed a scheme of formation of the bond between sapphire (alpha-Al_2O_3) and nickel alloys (fig. 3). The alloying elements, at the first moment of interaction, are distributed uniformly throughout the drop and then the atoms are segregated along the ceramic - metal interface, form new phases and then diffuse through the forming layer in the

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Table 2

Chemical Compatibility of Refractory Metals and Ceramic Materials /19/

Керамика	Метал	Результаты	Температура испытания, °C	Метод получения
Al_2O_3	Mo	Не реагирует	1432—1740	ГП
Муллит	Mo	»	1640	СВ
$ZrSiO_4$	Mo	»	1595	ГП
TiC	Mo	Реагирует	2315	ГП
SiC	Mo	»	2065	ГП
ZrO_2	Mo	Не реагирует	1600	ГП
MgO	Mo	»	1480	ГП
B_4C	Mo	Реагирует	1540	ГП
TaC	Mo	»	2000	ГП
H_fO_2	Mo	Не реагирует	1456	ГП
$SrZrO_3$	Mo	»	1100—2010	ГП
NdO_2	Mo	»	1540	ГП
Муллит	W	»	1640	СВ
ZrO_2	W	»	1600	ГП
MgO	W	»	1480	ГП
B_4C	W	Реагирует	1540	ГП
TaC	W	»	2000	ГП
TiC	W	»	1870	ГП
H_fO_2	W	Не реагирует	1455	ГП
$ZrSiO_4$	W	»	1650	ГП
$SrZrO_3 + MoSi_2$	W	»	1730	ГП
NdO_2	W	»	1540	ГП

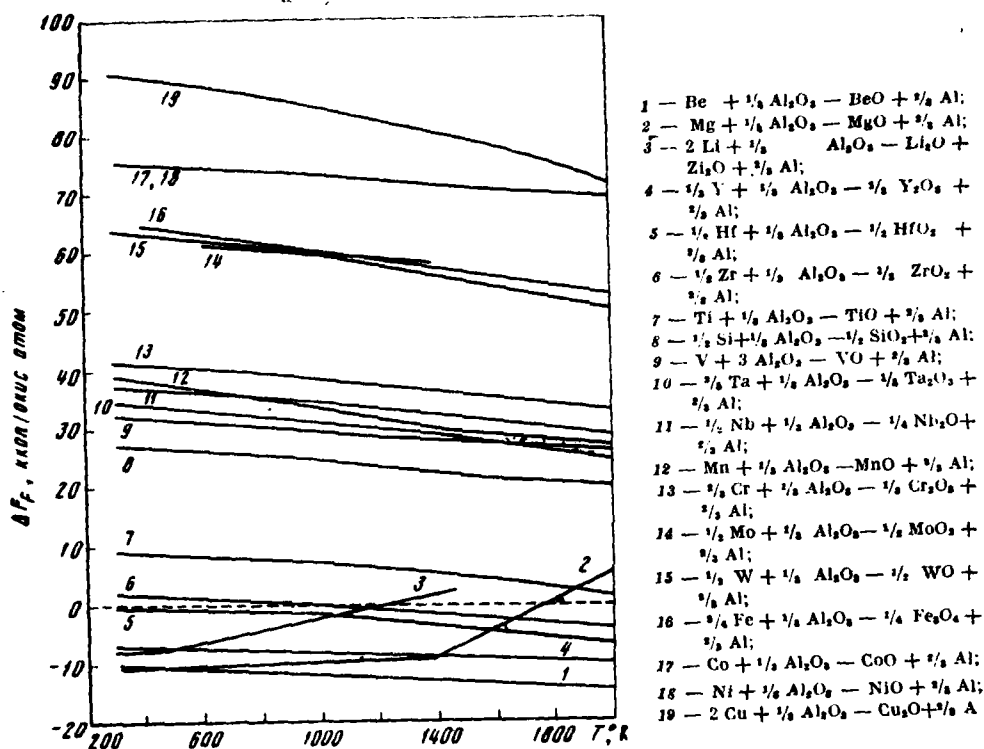Примечание. ГП — горячее прессование, СВ — спекание в вакууме.

1- Ceramics, 2- Metal, 3- Results, 4- Temperature of Test °C
5- Method of Production, 6- Does not react, 7- Reacts,
8- Note, 9- GP- Hot Forge Pressing, 10- Sintering in vacuum
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aluminum oxide. In the final stage of interaction, grooves arose along the edges of the drop which reconfirmed the interaction on the interfaces.

x-ray and fluorescence analysis of the zone of interaction permitted identification of phases which arise on the interface: titanium oxide with non-stoichometric composition,

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Cr_2O_3, zirconium oxide. It was established that, in case of
formation of zirconium oxide, oxygen was taken away from the
Al_2O_3 as manifested by the appearance of aluminum atoms in A
nickel fusion. If you assume that, during the presence of an
interaction reaction, the bond at the interface is strength-
ened then the elements are distributed in terms of effective-
ness as follows: zirconium, titanium, chromium.

Fig. 2. Temperature Dependence of the Interaction Reaction
Between Al_2O_3 and Me

1 — Be $+ \frac{1}{3} Al_2O_3 — BeO + \frac{2}{3} Al$;
2 — Mg $+ \frac{1}{3} Al_2O_3 — MgO + \frac{2}{3} Al$;
3 — 2 Li $+ \frac{1}{3} \quad Al_2O_3 — Li_2O + Zi_2O + \frac{2}{3} Al$;
4 — $\frac{1}{3} Y + \frac{1}{3} Al_2O_3 — \frac{1}{3} Y_2O_3 + \frac{2}{3} Al$;
5 — $\frac{1}{2} Hf + \frac{1}{3} Al_2O_3 — \frac{1}{2} HfO_2 + \frac{2}{3} Al$;
6 — $\frac{1}{2} Zr + \frac{1}{3} Al_2O_3 — \frac{1}{2} ZrO_2 + \frac{2}{3} Al$;
7 — Ti $+ \frac{1}{3} Al_2O_3 — TiO + \frac{2}{3} Al$;
8 — $\frac{1}{2} Si + \frac{1}{3} Al_2O_3 — \frac{1}{2} SiO_2 + \frac{2}{3} Al$;
9 — V $+ \frac{1}{3} Al_2O_3 — VO + \frac{2}{3} Al$;
10 — $\frac{2}{3} Ta + \frac{1}{3} Al_2O_3 — \frac{1}{3} Ta_2O_3 + \frac{2}{3} Al$;
11 — $\frac{1}{2} Nb + \frac{1}{3} Al_2O_3 — \frac{1}{4} Nb_2O + \frac{2}{3} Al$;
12 — Mn $+ \frac{1}{3} Al_2O_3 — MnO + \frac{2}{3} Al$;
13 — $\frac{2}{3} Cr + \frac{1}{3} Al_2O_3 — \frac{1}{3} Cr_2O_3 + \frac{2}{3} Al$;
14 — $\frac{1}{3} Mo + \frac{1}{3} Al_2O_3 — \frac{1}{3} MoO_3 + \frac{2}{3} Al$;
15 — $\frac{1}{2} W + \frac{1}{3} Al_2O_3 — \frac{1}{2} WO + \frac{2}{3} Al$;
16 — $\frac{3}{4} Fe + \frac{1}{3} Al_2O_3 — \frac{1}{4} Fe_3O_4 + \frac{2}{3} Al$;
17 — Co $+ \frac{1}{3} Al_2O_3 — CoO + \frac{2}{3} Al$;
18 — Ni $+ \frac{1}{3} Al_2O_3 — NiO + \frac{2}{3} Al$;
19 — 2 Cu $+ \frac{1}{3} Al_2O_3 — Cu_2O + \frac{2}{3} A$

However, if the force of interaction is evaluated by shearing
strength along the ceramic - metal interface, then these me-
tals are distributed in reverse order. This indicates that
intense interaction, while increasing the strength of the
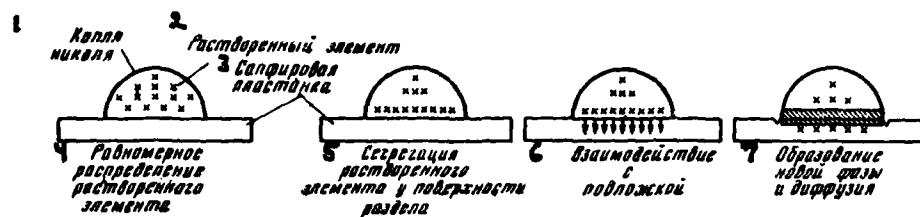bond, breaks down the ceramic backing.

Fig. 3. Diagram of the Rise of Bonding Between Metal and
  Sapphire Under the Effect of Alloying Additives in
  Metal /21/

1-Nickel drop, 2- Dissolved element, 3- Sapphire plate,
4- Uniform distribution of dissolved elements, 5- Segregation of dissolved element along interface, 6- Interaction with the backing, 7- Formation of a new phase and diffusion

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The effect of the opposed factors (increase of the bond between the ceramic and the metal and reduction of the strength of the ceramic as a result of interaction) is presented schematically in fig. 4. This drawing indicates that there exists some optimal intensity of interaction which ensures maximal strength of the bond. If the optimal intensity of interaction is exceeded, failure of the backing begins at low levels of strength.

The studies conducted indicated the necessity of limiting interaction to ensure minimum damage to the ceramic component of the composition. In the first approach, active alloying elements of the die have the same effect on ceramic fiber as that which occurs in the case of use of tungsten wire in a copper - tungsten composition. Evidently, in order to avoid this undesirable interaction, it is necessary, just as for metal - metal compositions, to conduct solid phase reactions of bonding of components and to introduce into the die ceramic fiber, precoated with metal. Thus, if the basic

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die alloy does not contain alloying additives in this case,
then the accumulation of soluble atoms along the interface
will be less which will reduce the intensity of interaction
between components in the process of forming a bond and af-
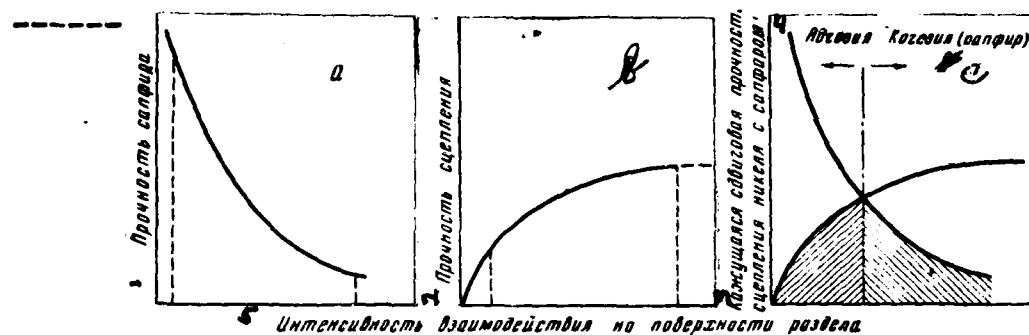ter prolonged operation of the composition.

Fig.4. Diagram of Reactions on the Interface of the Nickel -
Sapphire Composition on the Strength of the Composit-
ion /?/

a- reduction of strength of the backing; b- increase of the
strength of bonding; c- combined effect of these factors on
the strength of the bond
1- strength of sapphire, 2- strength of bonding, 3- apparent
shear strength of the nickel - sapphire bonding, 4- adhesion,
cohesion (sapphire), 5- intensity of interaction on the inter-
phase

The literature still contains only the results of a few
studies of the interaction of metal dies and ceramic fibers
of borides, nitrides, carbides with boric and carbon fibers.

Compatibility of components based on metal - boron,
metal - silicon carbide, metal - boron nitride, metal - bor-
on carbide and metal - silicon nitride $\wedge$ CONSTITUTION diagrams are
considered in study /1/. Boric fibers which possess inter-
esting mechanical properties and which are promising fibers
for reinforcement attracted attention to the study of the

interaction in boron – metal systems. The study of binary
metal – boron constitution diagrams led to the deduction con-
cerning the extremely high reaction capacity of boron, which
forms with metals many chemical compounds by virtue of which
production of thermodynamically stable metal – boron compo-
sites is extremely difficult. Systems in which interaction
is absent include Cu – B, Ag – B and Au – B systems (fig. 5).
The very absence of interaction in these systems permits the
recommendation of silver, in particular, as a barrier for
impeding the interaction in an Ni – B system. Most ideal is
the compatibility of components in a boron – silver system,
since, on the constitution diagram not only are bondings ab-
sent but there also is a place of immiscibility in the liquid
state. An Au – B system is similar to an Ag – B system and
therefore gold also may be used as a barrier layer. The Cu –
B constitution diagram is eutectic and some researchers ob-
served in a Cu – boron fiber composition transition zones in
the copper die. Experiments showed /20/ that, in Fe – boron
fiber, stainless steel – boron fiber, aluminum – boron fiber
compositions, even after soaking for one hour at $900^{\circ}C$, there
is formed,around the fiber, a 20 mk reaction zone consisting
mainly of borides of the metal. In the case of an aluminum-
boron composition, the reaction zone after interaction in a
vacuum consists of aluminum diboride (AlB_2), dodecoboride
(AlB_{12}) and an aluminum layer, saturated with boron. In air,
the boron is oxidized and B_2O_3 is formed. Coating boron fiber
by silicon carbide greatly reduces the intensity of the inter-
action reaction.

The interaction reaction between titanium alloy Ti8Al -
MolV and boron proceeds much more slowly than is the case
for aluminum (fig. 6). For comparison, the interaction be-
tween Tl6Al4v alloy and SiC is plotted in fig. 6.

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Nickel interacts intensely with boron, forming several compounds. The same such active interaction was observed in an Ni - Cu - B system (part of this diagram for the Cu - NiB_2 section is presented in fig. 7. A die (70% Ni, 30% Cu),reinforced by boron fiber was heated up at a rate of 50 degrees per minute in order to study the reaction in this system. A reaction with formation of nickel boride was observed at 600-700°. Copper was given off from the die in the process of this reaction. The presence of copper in the reaction zone had a harmful effect on the strength of the composition because of the more intensive weakening of the fibers. A study of the drop in strength of boron fibers, coated with nickel and subjected to prolonged annealing (1 day) at different temperatures up to 1000°C (fig. 8), showed that the abrupt weakening of the fibers (and, consequently, strong interaction) appears between 400 and 600°C. Barrier coatings of Ni_3Al, CoSi, FeSi which are in relative equilibrium with boron, are recommended for reducing interaction in metal - boron systems. Developers of composite materials placed great hopes on a metal - silicon carbide system since silicon carbide as a hardener (whiskers and fibers) is cheaper than boron and more creep resistant than it. However, a study of constitution diagrams of refractory metals - silicon - carbon showed that silicon carbide interacts actively with titanium, zirconium, niobium, tantalum, molybdenum and tungsten (fig. 9) /1/. It was shown also that, in many molten metals (iron, copper, gold, palladium, platinum), silicon carbide dissociates on free graphite and silicon with subsequent formation of silicides and carbides. Analysis of the constitution diagrams presented showed that thermodynamic compatibility in metal - silicon carbide systems is impossible. As concerns kinetic compatibility, it does take place in some cases as, for example: 1) the rate
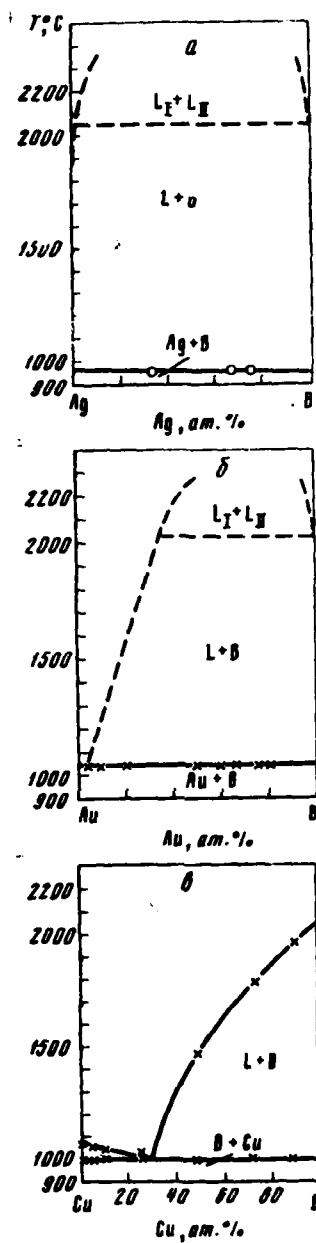
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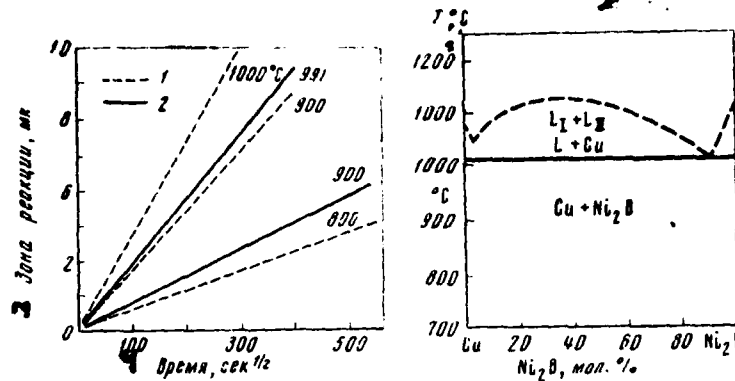Fig. 5. Ag - B (a), Au - B (b) and Cu - B (c) systems /1/

Fig. 6. Isothermic Growth of the Zone of Reaction in Ti 6 Al
4V-SiC; Ti 8Al 1Mo 1v-B Systems /20/

1- boron; 2- silicon carbide; 3- reaction zone, mk; 4- time,
seconds

Fig. 7. A Cu - Ni$_2$B System /1/

Fig. 8. Change of Strength of Boron
Fibers, Coated with Nickel
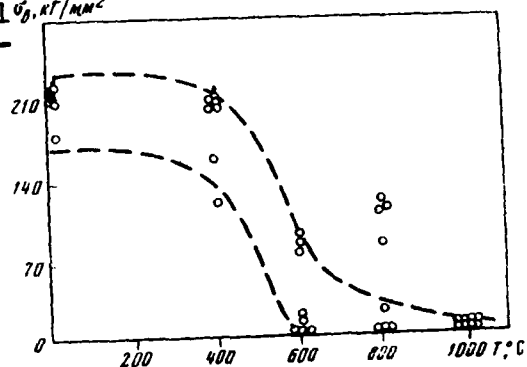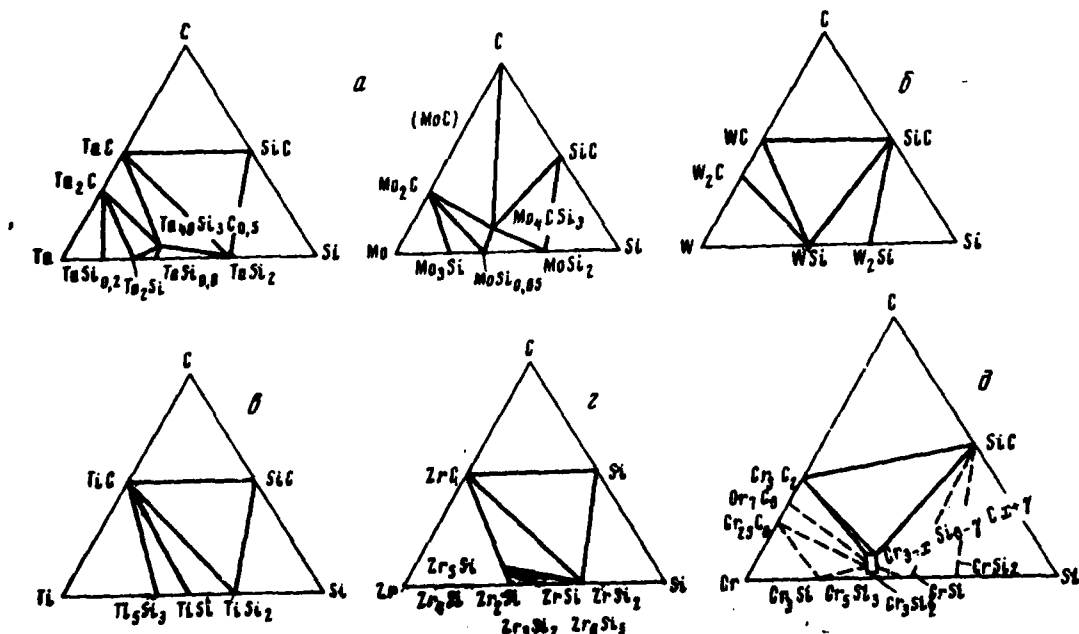After Annealing at Different
Temperatures For One
Day /22/

Fig. 9. Metal - Carbon - Silicon Systems /1/

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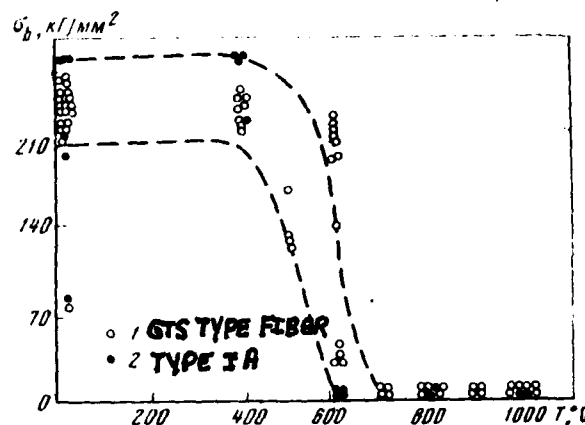$\sigma_b, \kappa\Gamma/MM^2$

Fig. 10. Change of Strength of Silicon Carbide, Coated by
Nickel After Annealing at a High Temperature For
One Day /22/

1- GTS type fiber; 2- IA type

of interaction in a titanium - silicon carbide system is in-
significant up to 1300°C; 2) a nickel - silicon carbide sys-
tem is stable up to 1000°C; the cause of this is unclear:
this stability may be explained by the presence of silicon
oxide being found on the silicon carbide surface or the
crystallographic orientation of the silicon carbide fibers
played a decisive role in this case.

According to other data, the interaction between nickel
and silicon carbide fibers begins to appear already after
prolonged soakings (1 day) at 500°C, leading to a drop in the
strength of the fibers ,which drops almost to 0 even at 700-
800°C (fig. 10)/22/. There is practically no interaction of
aluminum with silicon carbide right up to the fusion point.
Interaction, determined by a drop in strength of the fibers,
coated by aluminum was observed only after prolonged soakings
at 700°C /22/. During study of compatibility of silicon car-
bide with dies from nickel - chromium steels and alloys,great
reaction capacity also was revealed: silicon carbide reacted
with the die with formation of Cr_2C_3 + C and Si compounds and,

at high concentrations and more complex compounds: Ni_3Si +
Ni_5Si, ternary compounds.

On the basis of results obtained, the conclusion is made
/1/ that it is extremely difficult for silicon carbide to take
up a die which is not interacting with it. Exceptions, evi-
dently, are complex alloys which contain carbides and sili-
cides of metals in their composition. The structure of ter-
nary metal - N_2 - B systems showed (fig. 11)/1/ that boric
nitride - a potential reinforcing material -- but only in
specific temperature regions (up to 1327 $^\circ$C with tungsten,
in the 1227 - 1727° interval with chromium) located in equi-
librium with the metal. Borides of metals and nitrogen are
found in equilibrium with the metal in other temperature re-
gions. Nickel reacts with boric nitride in the solid-phase
region and the reaction proceeds with liberation of gaseous
nitrogen : when the composite material was heated to a tem-
perature at which fusion occurred around the fibers , it was
possible to observe,after cooling, a large number of pores in
the zone around the fibers. Analysis of the Ti - C - B, Mo -
C - B and Ta - C - B constitution diagrams (fig. 12) showed
/1/ that boric carbide is never found in equilibrium with
metal even in some limited temperature regions as in the case
of boric nitride.

Thus, on the basis of examination of the data of the lit-
erature and experiments conducted, the assumption is made/1/
that not one of the potential materials (boron, silicon car-
bide, boric nitride, boric carbide) is thermodynamically sta-
ble in metal dies used in technology. Interaction between
different ceramic fibers and metal dies were also studied in
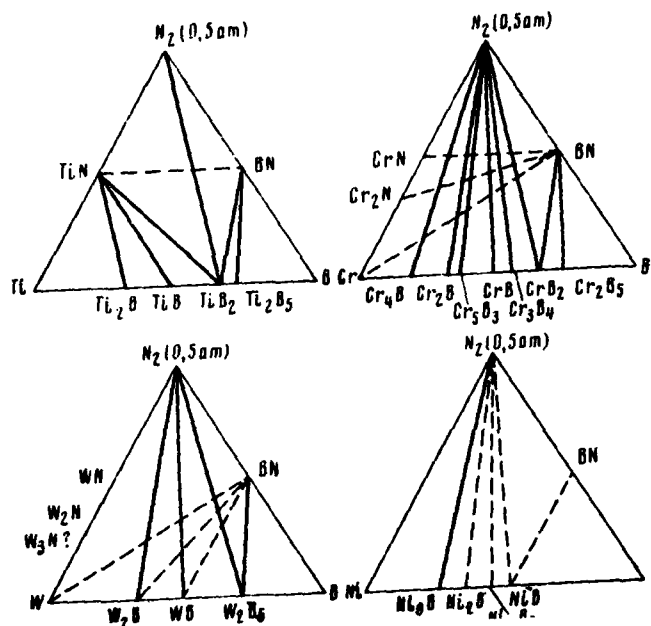detail in /14/.
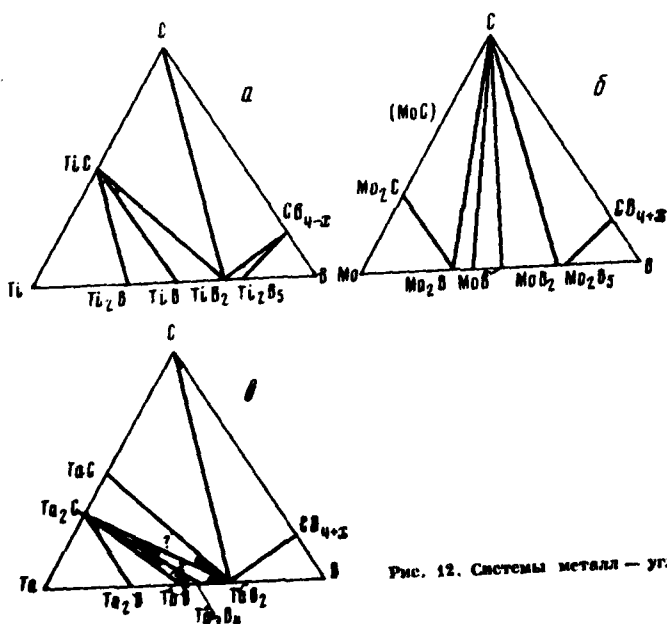
Fig. 11. Metal – nitrogen – boron Systems /1/

Рис. 12. Системы металл – углерод – бор /1/

Fig. 12. Metal – carbon – boron Systems /1/

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Interaction of 1)boron fibers with an aluminum die;
2) silicon carbide fibers with aluminum and nickel dies;
3) Al_2O_3 fibers with a nickel die has been studied. In these
experiments, the fibers, as a rule, were kept for a specific
time in contact with the molten metal and then the congealed
composition underwent additonal heat treatment. The inter-
mediate layer between components of the composition was
studied by means of preparing metallograohic specimens of the
composition by light and electron micoscopy methods and micro-
x-ray diffraction. The zone of interaction reached 5 mk in a
boron fiber - aluminum composition after a 15 minute stay of
the boron fibers in the aluminum melt at $680^{\circ}C$. Under these
same conditions, the zone of interaction in aluminum - sili-
con carbide compositions reached only 3 mk. The zone of in-
teraction was determined on the basis of electron microscopy
data and x-ray analysis (at a point) which fixes the change
of the elements (in this case B, Al, W - nucleus of boron
fibers and Si, C) along the cross section of the specimen.

Tables 3 -5 contain basic results of studies of the in-
teraction between components of compositions.

The studies indicated:

1) the comparative inertness of oxidic ceramic (Al_2O_3)
during interaction with a metal die;

2) the greatest interaction with a metal die was seen
for silicon carbide; the depth of interaction increased to
10 times and even 100 times in comparision with that for
Al_2O_3;

3) in some cases, whisker crystals, which have large
specific surface, were completely "dissolved" in the die;

Table 3. Interaction Between Silicon Carbide in Different Forms and Nickel Die

1- Material and primary source, 2- Die, 3- Temperature of the cast form, 4- State of the composite, 5- depth of penetration,mk, 6- maximum, 7- minimum, 8- average, 9- A massive bar of silicon carbide,d= 3 mm, 10- SiC whiskers (d=2mk) Thermokinetic Fibers Ltd., 11-SiC fiber (d = 100 mk) United Aircraft Corp., 12- SiC fiber (d=0.5 mm) Slase Development Ltd., 13- SiC fiber, d>0.28 mm, Fuller Rand, 14- Pure Nickel, 15- Nimokast, 16- Nickel, 17- After casting, 18- Casting, 19- Annealing at 1100°C, 50 hr. 20- After casting, annealing at 1100°C, 50 hr, 21- Hot pressure forging Whiskers fractured, at 1000°C, 22- After casting, 23- Interaction throughout the diameter of the fiber, 24 - the same

1 Материал и перво-источник его	2 Матрица	3 Темпера-тура литейной формы	4 Состояние композита	Глубина проникно-вения, мк		
				6 max	7 min	8 сред-няя
9 Массивный стержень карбида кремния, d = 3 мм	14 Чистый никель	20	17 После литья	1	1	1
		800	19 Литье	80	40	60
	15	1200	»	120	110	100
	Нимокаст	20	»	1	1	1
	713С	800	»	110	70	80
		1200	18 »	200	100	140
		20	19 Отжиг при 1100° C, 50 час	40	10	15
	15 Нимокаст	20	20 После литья, отжиг при 1100° C, 50 час	1	1	1
	75			20	16	18
10 Усы SiC (d = 2 мк) компании Thermokinetic Fibers LTD	16 Никель	—	21 Горячее прессование при 1000° C	Усы разрушались		
	15 Нимокаст 75	20	22 После литья	»		»
11 Волокна SiC (d = 100 мк) компании United Aircraft Corporation	14 Чистый никель	20	» »	1	—	1
	15 Нимокаст 75	800	» »	23 Взаимодействие по всему диаметру волокна		
	16 Нимокаст 713С	800	» »	24 То же		
12 Волокна SiC (d=0,5 мм) компании Slase Development LTD	Нимокаст	200	» »	40	20	30
	15 313	800	» »	160	120	140
	Нимокаст 75	20	» »	63	63	140
13 Волокна SiC диаметром более 0,28 мм компании Fuller Rand	Нимокаст 713С	800	» »	160	120	140

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Table 4

Interaction Between Silicon Nitride and Alloys Based on Nickel

Материал и первоисточник его	Матрица	Состояние композиции	Глубина проникновения, мк		
			max.	min	средняя
Горячепрессованный поликристаллический стержень Si₃N₄	Нимокаст 75	После литья,	4	0,7	2
		отжиг 20 час при 1100° С	—	—	30
		Отжиг 50 час при 1100° С	—	—	50
	Нимокаст 258	После литья,	6	1,6	4
		отжиг 50 час при 1100° С	40	23	32
Усы Si₃N₄	Коммерческий никель	Горячее прессование 0,3 час при 1000° С	0,2	Усы не разрушались	
		Горячее прессование 50 час при 1000° С		То же	

1- Material and its primary source, 2- Die, 3- State of the composition, 4- Depth of penetration, mk, 5- maximum, 6- Minimum, 7- Average, 8- Hot pressure forged polycrystalline bar of Si_3N_4, 9- Si_3N_4 whiskers, 10- Nimokast, 11-Commercial nickel, 12- After casting, annealing 20 hr at 1100°C, 13- Annealing 50 hr at 1100°C, 14- After casting, annealing 50 hr at 1100°C, 15- Hot pressure forging 0.3 hr at 1000°C, 16- Hot pressure forging 50 hr at 1000°C, 17- Whiskers were not fractured, 18- the same

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4) the intensity of interaction depended on the form of the nickel die, the structure and degree of purity of the ceramics.

Comparatively little is known about the interaction of carbon fibers with metals which are promising for strain hardening. Four kinds of interactions of carbon fiber and metal are possible. They are 1) formation of carbides, 2)oxidation, 3) recrystallization; 4) formation of solid solutions.

A detailed study of the interaction between carbon fibers and different metals is presented in /22/. The study was conducted as follows: graphite fibers ,produced by the "Rolls Royce" firm and having an initial mean strength of 190 kg/mm^2 and modulus of elasticity of 42,000 kg/mm^2, were coated with material of the die being studied. An aluminum coat was applied by vacuum metal spray coating; nickel, cobalt, chromium, platinum and copper was applied to carbon fibers by the electrolytic method. After application of the coatings, the fibers were gathered into a bundle ($\sim$ 15 fibers) and annealed in a vacuum ($2 \cdot 10^{-4}$ mm mercury column) for different durations (up to 200 hrs) and at different temperatures (up to 1100°C). interaction occurring at the fiber – coating interface was studied by several methods: metallographic analysis of transverse metallographic specimens of fibers and composites with graphite fibers, study of the surfaces of fibers in an electron scan microscope, an x-ray study of changes of the structure in carbon fibers and mechanical properties of the fibers.

Measurements of the strength of the fibers before and after heat treatment were conducted by determination of the strength of the microcomposite: the graphite fibers bundles were transformed, in a special assembly, into a thread of the microcomposition consisting of grahite fibers and an expoxy resin with known properties. From determination of the strength of this microcomposite, we calculated the strength of the components of its carbon fibers. The measurement of the strength was the basic criterion of the course of interaction between the components. Figure 13 shows the change of strength of graphite fibers not coated by metal. No marked reduction of strength of the fibers was noted right up to 1100°C. Results of change of strength of aluminum coated graphite fibers after a one-day soaking at different temperatures are shown in fig. 14. Prolonged soaking (one week)

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of aluminum coated fibers at 500°C did not cause reduction of strength which makes it possible to assume the stability of graphite fibers in aluminum dies and under loading during prolonged operation in a temperature region up to 500°C. Annealing aluminum coated graphite fibers for one day at 600°C led to formation of aluminum carbide (Al_4C_3) and reduction of strength. Tests above 700°C were not conducted since the molten aluminum had a tendency to collect on the fibers in drop form.

Table 5. Interaction Between Aluminum Oxide (Different Forms) and Dies on a Nickel Base /14/

1 Материал и его первоисточник	2 Матрица	3 Состояние композита	4 Глубина проникновения, мк		
			5 max	6 min	7 средняя
8 Сапфировый стержень (монокристалл)	Нимокаст 258	13 После литья отжиг 100 час, при 1100° С	2	—	2
d = 1,5 мм компании Salford Electricl Instruments LTD	Нимокаст 713С	14 Отжиг 100 час при 1100° С	3	1	2
9 Поликристаллический Al₂O₃	Нимокаст 258	Отжиг 100 час при 1100° С	3	1	2
компании	Нимокаст 75	15 То же	3	1	2
Morgan Crucibl Co	Нимокаст 713С	»	3	1	2
10 Стержень Al₂O₃	Нимокаст 713С	»	3	2	2
d = 0,25/0,75 мм компании Morganite Research and Development					
11 Монокристаллическое волокно компании Tyco Laboratories LTD	Нимокаст 713С	Отжиг 100 час при 1100° С	3	2	2
		16 Отжиг 300 час при 1100° С	5	3	3
		17 Отжиг 100 час при 1200° С	6	3	4

1- Material and its primary source, 2- Die, 3- State of Composite, 4- Depth of penetration, mk, 5- Maximum, 6- Minimum, 7- Average, 8- Sapphire bar (single crystal) d=1.5 mm, Salford Electrical Institute Ltd, 9- Polycrystalline Al_2O_3, Morgan Crucible Co., 10- Al_2O_3 bar, d=0.25/0.75 mm, Morganite Research and Development Company, 11- Monocrystalline fiber

Tyco Laboratories Ltd, 12- Nimokast, 13- After casting and annealing 100 hrs at 1100°C, 14- Annealing 100 hrs at 1100°C, 15- the same, 16- Annealing 300 hrs at 1100°C, 17 - Annealing 100 hrs at 1200°C

Note. In all cases, the temperature of the foundry form was 800°C.

Fig. 13. Change of Strength of Graphite Fibers After Annealing For One Day at High Temperature /22/
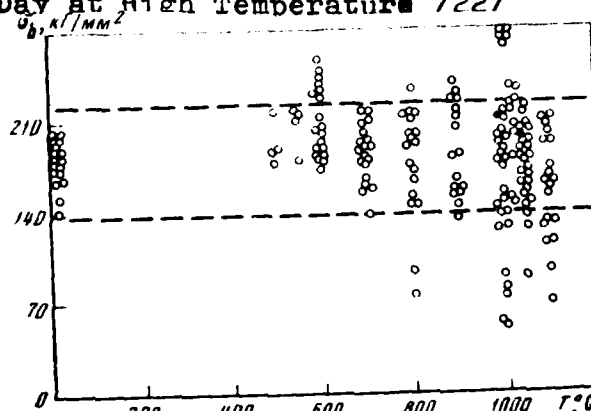
Fig. 14. Change of Strength of Graphite Fibres Coated by Aluminum, After Annealing For One Day At High Temperature/22/
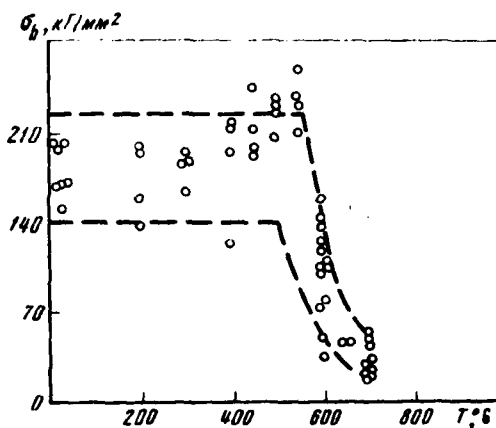
Figure 15 presents results of reducing strength after
annealing nickel coated graphite fibers. The effect of the
duration of annealing at 1000°C on the strength of the fibers is shown in fig. 16.

Nickel coated graphite fibers are recrystallized as a
result of prolonged annealing (one day at 1100°C or four
days at 1000°C) and their structure begins to resemble the
structure of natural graphite. In this case, the recrystal-
lization of graphite fibers was characterized by 1) the
appearance of additional lines on the roentgenogram; 2) by a
growth of grain size which is manifested through an increase
of the definition of the ghosts; 3) by a change in the de-
gree of orientation of crystals relative to the axis of the
fiber. Figure 16 shows the correlation between the drop of
strength of fibers and the recrystallization process. The
effect of the influence of nickel on recrystallization of
graphite fibers is associated with the acceleration of dif-
fusion processes. The effect of recrystallizaion of graph-
ite fibers in the presence of nickel was further studied on
a graphite fiber - nickel die composition, produced by hot
pressure forging (T=900°C, 1 hour, vacuum, pressure of 1.4
kg/mm²) of nickel coated fibers.

Figure 17 shows changes of structure of this composit-
ion after annealings. This experiment revealed a good con-
formity between recrystallization of nickel coated fibers
and recrystallization of fibers in the composite.

Figure 18 shows the dependence of changes of strength
of cobalt coated graphite fibers after different annealings.
The mechanism of effect of cobalt on reduction of strength
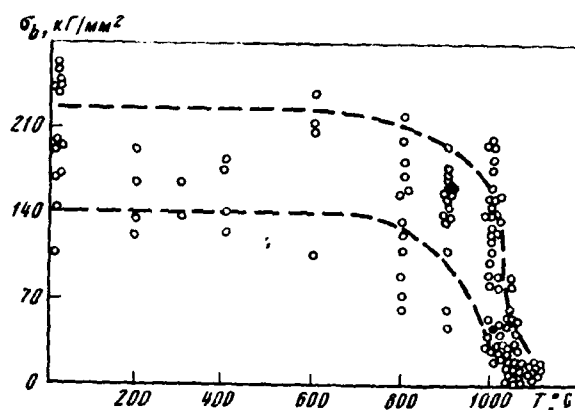of fibers is the same as that for nicekl: cobalt causes

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Fig. 15. **Change** of Strength of Nickel Coated Graphite Fibers
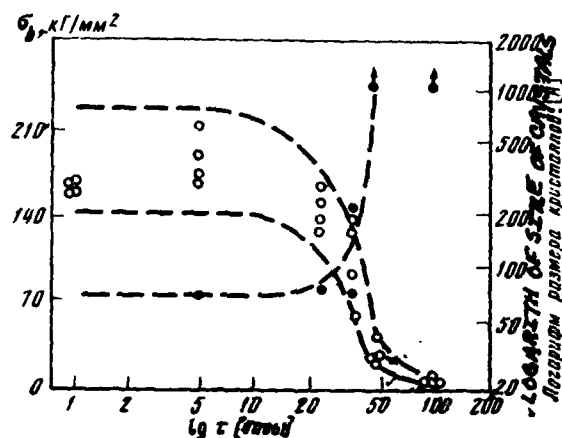**After** Annealing For One Day at High Temperature /??/

Fig. 16. Change of Strength and Grain Size of Nickel Coated
Graphite Fibers After Annealing For Different
Durations at 1000°C /??/

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recrystallization of the graphite which is characterized by
an increase of dimensions of the crystals in the fibers.
Figure 19 shows the interaction of the graphite fibers with
the nickel - chromium layer of the coating in the annealing
process. A drop in strength was observed even at 600°C and
was connected with the formation of chromium carbides (Cr_3C)
i.e. the mechanism of deterioration of the strength of the

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fibers resembles the mechanism of interaction of aluminum and graphite fibers. Graphite fibers coated with platinum and copper, after annealing for a day at 800-900°C, also revealed a drop in strength although no visible signs of interaction between the metal and the graphite were observed.

As was shown above, upon contact of carbon fibers with smelted aluminum at 600-700°C, there occurs oxidation of the fibers and formation of carbides /22/ with an Al_4C_3 structure. It was demonstrated experimentally /23/, that a stay in smelted aluminum for five minutes during production of compositions of carbon fibers led to a formation of carbides and reduction of the mechanical properties of the composition. However, the stay of carbon fibers, coated with a special metallic coating for improving the wetting, for several seconds in molten aluminum (680°C) did not lead to a formation of carbides, reducing the strength of the composition. The activity itself of carbon in respect to metals reduces potential possibilities of metal - carbon fibers compositions. However, with proper control of the interaction process, there is a possibility of producing metal - carbon compositions by a plasma method /24/. A study of the conditions of interaction during plasma deposition between graphite and refractory metals (Mo, Ti, Nb) indicated that a firm bond is formed with the appearance of chemical interaction with formation of carbides - Nb_2C, NbC, TiC, Mo_2C. The process of plasma deposition of the die permits flexible regulation of the intensity of interaction and, consequently, regulation of the quality of future compositions.

Since glass and quartz fibers possess great strength and are used extensively in technology, the development of metal compositions, reinforced by these fibers, was extremely tempt-
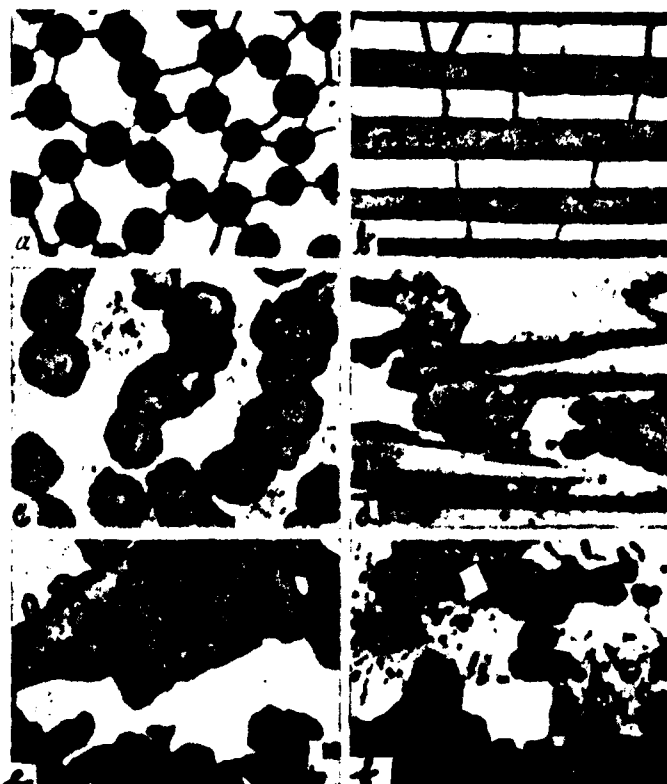
63

Fig. 17. Recrystallization of Graphite Fibers in a Nickel-
Graphite Composition
a,b- After hot pressed forging; c,d -after annealing, 4 days,
$1000^\circ C$; e,f- the same, $1100^\circ C$ /22/

----

ing. During creation of these compositions it is necessary
to watch carefully that the interaction reaction between the
components along the surface of the interface did not lower
the initial strength of the fibers. In order to reduce the
interaction reaction in the process of manufacturing aluminum-
quartz fiber compositions, the quartz fiber is precoated with
aluminum /3/. Upon controlling the interaction reaction along
the interface or, more precisely, ensuring its uniformity,
it was possible to produce an Al - SiO_2 fiber with strength
of 105 kg/mm^2.

However, technological difficulties in the production of this composition and devitrification of the quartz during interaction with aluminum, causing a loss of strength, limited the possibilities of use of this composition.

Considering the interaction of components in composite materials as a whole, we must emphasize that the technology of production of fibrous compositions largely determines the intensity of passage of reactions on the interfaces.

An increase of the temperature of deposition led to a marked increase of interaction/3/. Coating tungsten fiber with cobalt alloy L-605 (coating temperature is higher than that in compositions with a copper die) facilitated the dissolution and recrystallization of a significant part of the fibers which led to a marked reduction of strength of the composition. A change of the technology of production of the composition, for example, a change for a given composition from a technology of deposition to a technology of cold forge pressing and sintering /2/ leads to reduction of the rate of diffusion and intensity of interaction. Other researchers have used successfully sintering of components of compositions in the solid state. Kratchli /13/ produced an aluminum – stainless wire composition by hot forge pressing at 500°C. The comparatively low forging temperature did not cause significant interaction of the components of the composition. The powder metallurgy method was used for production of a Ti – 6Al – 4V – molybdenum wire composition.

The low temperature of processing (980°C) ensured low interaction on the fiber – die interface and, consequently, significant strain hardening of THE ore.
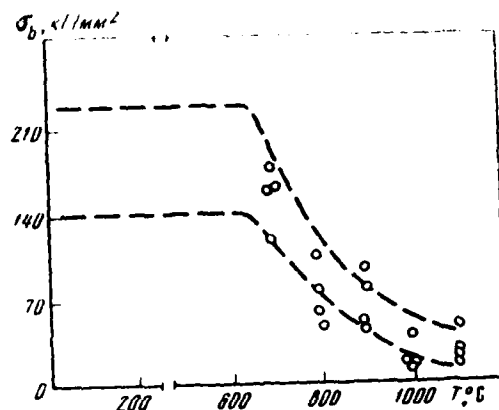
65

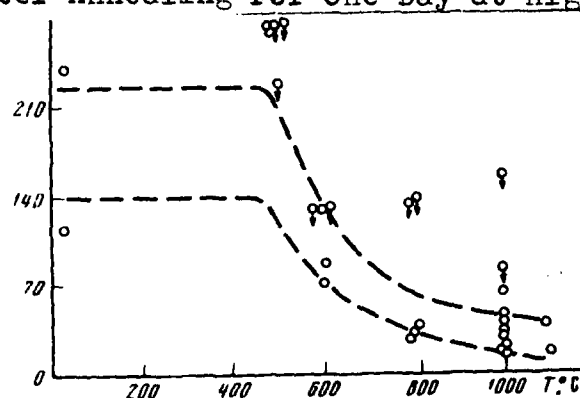Fig. 18. Change of Strength of Cobalt Coated Graphite Fibers After Annealing for One Day at High Temperature /22/

Fig. 19. Change of Strength of Graphite Fibers, Coated by Nickel – Chromium Alloy After Annealing For One Day at High Temperature /22/

Substition of liquid-phase reactions for solid-phase reactions during production of composites is the **first means** of reducing technological interaction between the die and the fibers. A **second means** of reducing the harmful effect of interaction at the interface of compositions is the creation of diffusion barriers. These barriers have no significant effect upon prolonged, service interaction but may reduce technological interaction significantly. The appli- of chromium coatings to tungsten fibers, even in case of liquid-phase reactions, led to reduction of interaction be-
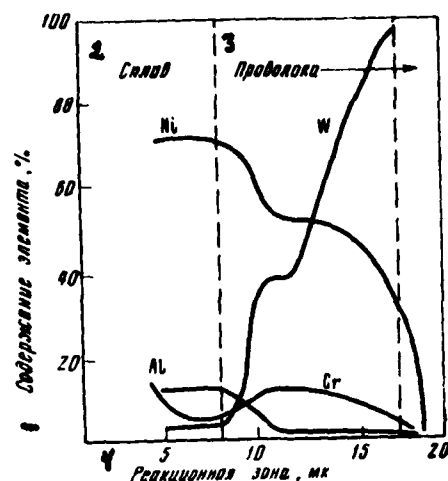
Fig. 20. Interaction Between Tungsten Wire and Nimokast 713C
Alloy After Soaking 600 Hours at 1100°C /14/

1. Content of elements, 2- Alloy, 3- Wire, 4- Reaction zone,
   mk

————————————

tween the fiber and the die in the process of producing the
composition. We reinforced stainless steel dies with tung-
sten wires coated by cobalt (18 % by volume). This rein-
forcement greatly increased the strength of the die from 28
to 41 kg/mm^2 at the same time as the strength of the die af-
ter reinforcement at the same fraction volume with tungsten
wire but without coating was increased only to 6 kg/mm^2. The
coating, reducing the brittleness of the peripheral parts of
the fibers, led to an increase of strength of the die. A
third means of reducing interaction intensity is directional
alloying of the metal die which makes the dies inert in re-
spect to the fiber material. There is a report that, in some
cases, during creation of nickel alloy - tungsten wire com-
positions, it was possible to so select the composition of
the die that it possesses almost no technological or service
interaction with the fiber material. Thus, for example, fig-
ure 20 presents results of a study by the method of micro-
x-ray analysis of the interaction between Nicrome alloy and
tungsten wire /14/. It is seen that, even after hundreds of

hours of soaking, the zone of interaction is several microns.
Interaction reactions on interfaces of composite materials
have significant effect on the properties of compositions
and require fixed attention. This interaction must be stud-
ied both on models and directly on different types of compo-
site materials. The necessity for a study of interaction re-
actions directly on composite materials is associated with
peculiarities of interfaces in fibrous compositions - defor-
mation of layers of the die bordering the fiber, development
of the fiber surface, specific structure of the fiber etc.
Only fundamental studies of interaction will permit creation
of a reliable technology of production of composite materials
and prediction of potential possibilities of different compo-
sitions.

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